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# IN-PLACE ELECTRICALLY HEATED REGENERATION OF VAPOR-PHASE ACTIVATED CARBON

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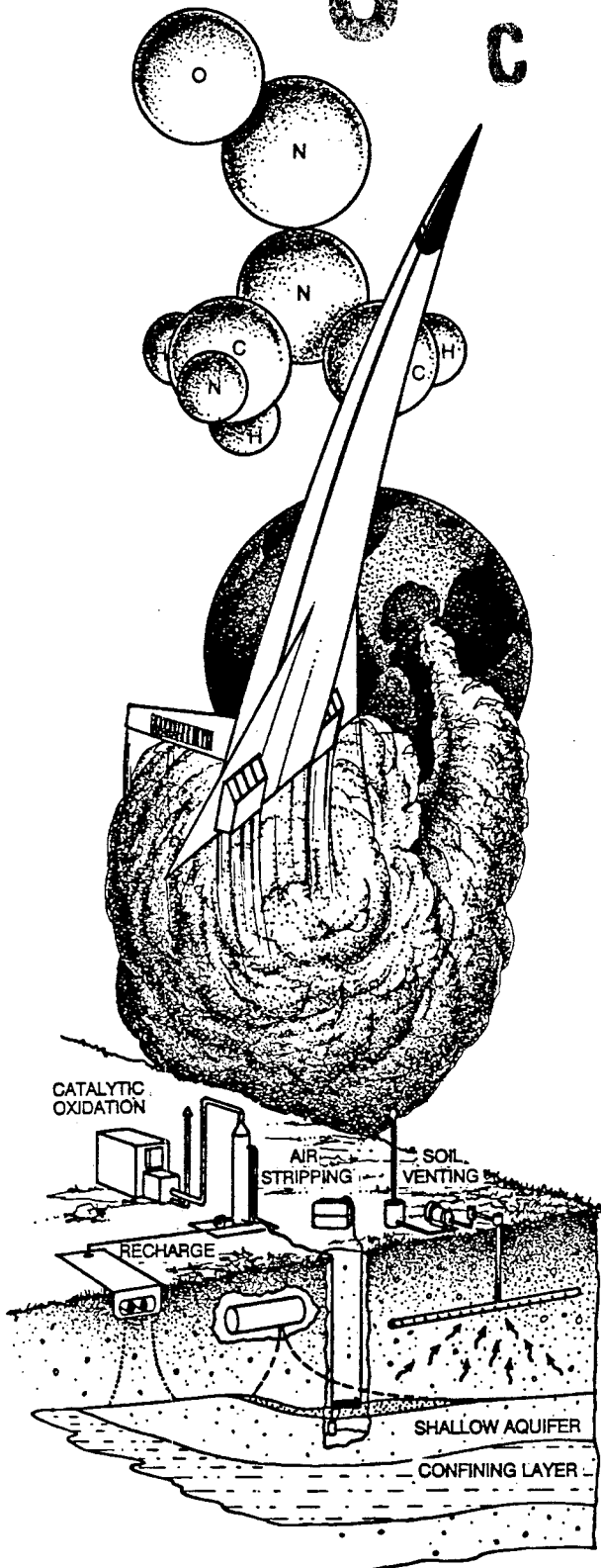
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FINAL REPORT

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**ENVIRONICS DIVISION**  
Air Force Engineering & Services Center  
ENGINEERING & SERVICES LABORATORY  
Tyndall Air Force Base, Florida 32403



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6 September 1990

Final Technical Report  
Contract No. F09635-89-C-0345  
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Prepared for:

Headquarters, Air Force Engineering and Services Center  
Directorate of Engineering and Services Laboratory  
Tyndall AFB, FL 32403-6001

Project Officer: Dr. Alexandre R. Tarsey (AFESC/RDVW)

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<p>The Air Force is using the air stripping process to remove a variety of volatile organic contaminants from groundwater. Vapor-phase granular activated carbon (GAC) has proven successful in treating the contaminated air stream emanating from air stripping columns, but conventional regeneration methods are expensive, require transfer of the carbon, and degrade the carbon. The objective of this experimental program was to determine the feasibility of using electrically-heated processes for the in-place regeneration of vapor-phase GAC.</p> <p>Two processes were considered, namely microwave heating and electric resistance heating using the carbon itself as the resistance. The microwave heating equipment used gave nonuniform heating over the length of the carbon column and was not pursued further. Electric resistance, or Rintoul, heating is a commercially proven process that has been applied to the high-temperature regeneration of liquid-phase GAC in special purpose furnaces. The experimental program demonstrated that the Rintoul process can be efficiently applied to low-temperature, low-energy, in-place regeneration of vapor-phase GAC. Repeated loading and regeneration of the carbon column showed no loss of adsorption capacity. Two methods of collecting TCE in a concentrated form were also successfully tested. Both processes involved recovery of the TCE in a condensate.</p> <p>Energy costs for the Rintoul process were conservatively estimated to be about half, but could be as low as one-twentieth, that for steam regeneration. All other costs, including labor, tail-gas purification, maintenance, and capital are also expected to be less than those for steam.</p> <p>Apart from its favorable costs, other major advantages of Rintoul regeneration are the ease of retrofitting, its application to small systems, and its portability. Thus, in addition to its use in air stripping for cleaning up contaminated groundwater, Rintoul regeneration can be used in applications where air purification by GAC and/or recovery of the volatile organic contaminant(s) are required.</p>					
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## EXECUTIVE SUMMARY

The Air Force is using the air stripping process to remove a variety of volatile organic contaminants from groundwater. Use of this process removes the contaminant from the liquid stream and discharges the pollutant into the air. Vapor-phase granular activated carbon (GAC) has proven successful in treating the air stream, but conventional regeneration methods are expensive, require transfer of the carbon, and degrade the carbon. The objective of this experimental program was to determine the feasibility of using electrically-heated processes for the in-place regeneration of vapor-phase GAC.

Two processes were considered, namely microwave heating and electric resistance heating using the carbon itself as the resistance. The microwave heating equipment used gave nonuniform heating over the length of the carbon column and was not pursued further. Electric resistance, or Rintoul, heating is a commercially proven process that has been applied to the high-temperature regeneration of liquid-phase GAC in special purpose furnaces. The experimental program demonstrated that the Rintoul process can be efficiently applied to low-temperature, low-energy, in-place regeneration of vapor-phase GAC.

Two methods of collecting TCE in a concentrated form were successfully tested. Both processes involved recovery of the TCE in a condensate. Energy costs for the Rintoul process were conservatively estimated to be about half, but could be as low as one-twentieth, that for steam regeneration. All other costs, including labor, tail-gas purification, maintenance, and capital are also expected to be less than those for steam.

Apart from its favorable costs, other major advantages of Rintoul regeneration are the ease of retrofitting, its application to small systems, and its portability. Thus, in addition to its use in air stripping for cleaning up contaminated groundwater, Rintoul regeneration can be used in applications where air purification by GAC and/or recovery of the volatile organic contaminant(s) are required. The process can also be used at hazardous waste sites to clean contaminated air from in situ vacuum collection processes.

## PREFACE

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This report documents work carried out between 11 July 1989 to 11 April 1990. The Air Force Project Officer was Dr. Alexandre R. Tarsey (HQ AFESC/RDVW).

The authors acknowledge the assistance and contributions of Professor R. H. Ma, Worcester Polytechnic Institute, Worcester, Massachusetts.

This report has been reviewed by the Public Affairs Office (PA) and is releasable to the general public, including foreign nationals.

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## TABLE OF CONTENTS

Section	Title	Page
I	INTRODUCTION.....	1
	A. OBJECTIVES .....	1
	B. BACKGROUND.....	3
	1. Air Stripping.....	3
	2. Stripper Off-Gas Treatment.....	4
	3. Stripper Off-Gas Heating.....	6
	C. SCOPE.....	7
II	ACTIVATED CARBON REGENERATION.....	8
	A. BACKGROUND.....	8
	B. THE RINTOUL PROCESS.....	10
	C. MICROWAVE REGENERATION.....	11
III	EXPERIMENTAL INVESTIGATION.....	15
	A. EXPERIMENTAL EQUIPMENT.....	15
	1. Selection of GAC and VOC.....	15
	2. Flow System.....	20
	B. GAS CHROMATOGRAPH TCE ANALYSIS.....	25
	C. RESULTS AND DISCUSSION.....	31
	1. Preliminary Testing.....	31
	2. Adsorption Tests.....	35
	3. Regeneration Tests.....	46
	4. Condensate Collection.....	56



TABLE OF CONTENTS  
(CONCLUDED)

Section	Title	Page
IV	COST ANALYSIS.....	59
	A. BACKGROUND.....	59
	B. ELECTRICAL AND THERMAL ENERGY.....	61
	C. ENERGY COSTS.....	62
	1. Rintoul Regeneration.....	62
	2. Steam Regeneration.....	64
	D. COST COMPARISON.....	65
	1. Regeneration.....	65
	2. Humidity Control.....	67
	3. Tail-Gas Treatment.....	68
	4. Microwave Regeneration.....	68
	5. Other Processes.....	70
V	CONCLUSIONS.....	71
VI	REFERENCES.....	73

# LIST OF FIGURES

Figure	Title	Page
1	Vapor-Pressure Curve for TCE.....	19
2	Schematic Diagram of Experimental Flow System.....	21
3	Breakthrough Curve for GAC.....	34
4	Breakthrough Curves for GAC with Rintoul Regeneration.....	38
5	Variation of GAC Capacity with Influent TCE Concentration.....	43
6	Ratio of Dry Air to Humidified Air Capacities vs. Influent TCE Concentration.....	43
7	Capacity of GAC Regenerated by Rintoul Heating.....	45
8	TCE Concentration During Regeneration.....	48

# LIST OF TABLES

Table	Title	Page
1	ESTIMATED ENERGY COSTS FOR REGENERATING 1000 KG CARBON....	12
2	VOCS THAT CAN BE EASILY REMOVED BY AIR STRIPPING.....	16
3	PHYSICAL PROPERTIES OF CALGON BPL CARBON.....	18
4	CHEMICAL PROPERTIES OF TRICHLOROETHYLENE (TCE).....	18
5	ANTOINE COEFFICIENTS FOR TCE.....	18
6	TCE VAPOR PRESSURE AND CONCENTRATION VS. TEMPERATURE.....	19
7	TCE GAS CHROMATOGRAPH STANDARDS AT ROOM TEMPERATURE.....	28
8	GAS CHROMATOGRAPH ANALYSIS OF FLOW SYSTEM AT ROOM TEMPERATURE.....	29
9	COMPARISON OF GAC CAPACITIES.....	34
10	SUMMARY OF ADSORPTION BREAKTHROUGH DATA.....	36
11	SUMMARY OF RINTOUL HEATING REGENERATION DATA.....	52
12	MEASURED TEMPERATURES IN CARBON BED DURING REGENERATION...	55

LIST OF TABLES (CONCLUDED)

Table	Title	Page
13	POWER INPUT TO CARBON BED DURING REGENERATION.....	56
14	CARBON BED TEMPERATURES VS. AIR TEMPERATURES.....	57
15	STEAM RATES FOR VOC REGENERATION.....	64
16	COMPARISON OF RINTOUL AND STEAM REGENERATION COSTS.....	69



## SECTION I

### INTRODUCTION

#### A. OBJECTIVES

The objective of this experimental investigation was to examine relatively low temperature granular activated carbon (GAC) regeneration based on electrical heating in terms of efficacy and economics. Two methods of electrically heating the carbon were studied:

- o Electric resistance heating, also known as Rintoul heating, using the carbon itself as the resistance
- o Microwave heating.

Specific objectives that pertain to regeneration were to:

- o Determine the optimum temperature at which the carbon can be efficiently regenerated.
- o Estimate the irreversible losses in carbon activity after repeated regenerations.
- o Determine the rate of desorption.
- o Determine the purge gas volume and time required for regeneration.
- o Estimate carbon losses when air is the purge gas
- o Determine the power requirements for regeneration.

Specific objectives pertaining to carbon capacity were to:

- o Investigate the effect of low-level heating during adsorption.
- o Determine the carbon capacity as a function of the relative humidity of the contaminated off-gas.
- o Determine the loss of carbon capacity over several loading and regeneration cycles.

The viability of the electric processes will ultimately depend on their cost, relative to established regeneration processes such as steam stripping. Specific objectives pertaining to the economics of the process were to:

- o Estimate the operating costs for regeneration by Rintoul heating and microwave heating.
- o Compare the cost of regeneration by steam with electrical heating.

During the course of the program, an additional objective was added to the scope of work, that of selecting a process to either destroy or recover chlorinated hydrocarbons given off during regeneration of the granular activated carbon by electric resistance heating. Laboratory tests were carried out to demonstrate feasibility of two selected processes.

The primary objectives of the program pertain to the use of in-place electrical heating as a means of reducing the regeneration costs. The use of this method as a means to provide low-level heating to increase the GAC capacity during adsorption is an added benefit, but is only a secondary objective.

## B. BACKGROUND

### 1. Air Stripping

Air stripping is one of the simplest and most economical processes for the removal of volatile organic chemicals (VOCs) from contaminated water. The reliability and effectiveness of air stripping on a large scale was first demonstrated at the 15-million gallon per day "Water Factory 21" project (Reference 1). However, stripping technology is equally effective for small-scale operations, where considerations of minimal equipment requirements and ease of operation are as important as low operating cost.

Both aqueous-phase granular activated carbon adsorption and air stripping have been specified by the United States Environmental Protection Agency as being "best available technology" (BAT) for control of VOCs from contaminated groundwater (Reference 2). Air stripping is about an order of magnitude less expensive than aqueous-phase adsorption GAC, and therefore has been the process of choice in cases where nonvolatile organics are not present (Reference 3). In cases where heavier organics are present, air stripping may still be used as a pretreatment to reduce overall costs as this extends the capacity of the aqueous-phase GAC and prevents premature breakthrough of the volatile components which are generally less strongly adsorbed.

The United States Air Force is using the air stripping process for cleaning groundwaters that have been accidentally contaminated with industrial solvents at various bases (References 3 and 4). Many of the installed systems are for the control of trichloroethylene (TCE), but contamination with tetrachloroethane (TTCE), tetrachloroethylene (PCE), toluene, benzene and n-pentane has also occurred. Other pollutants commonly found in contaminated groundwaters in the United States include motor fuels of all kinds, cis-1-2-dichlorethane, ethylbenzene, and the xylenes.

With the increased deployment of air stripping, the practice of simply discharging the stripper off-gases to the atmosphere without purification has become a cause for concern (Reference 5). Some states have already prohibited this practice, anticipating that discharge of contaminated off-gases will soon



be regulated at the Federal level. Off-gas purification equipment may have to be retrofitted to existing air strippers.

The added step of treating the stripper off-gases significantly increases both the cost and the complexity of air stripping. Unless these costs can be reduced, other technologies might prove to be more cost-effective for new installations. Consequently, considerable effort has been directed at developing cost-effective off-gas treatment technologies.

## 2. Stripper Off-Gas Treatment

Vapor-phase GAC adsorption of the stripper off-gas is one suitable process for treating stripper off-gases. This technically proven process has been used in the solvent-recovery industry (Reference 6) and other industries where volatile organics are released into the air. Gas-phase adsorption is very effective and economical compared to liquid-phase adsorption for the following reasons:

- o Adsorption kinetics for gases are faster than for liquids
- o Organic concentrations are higher in the stripper off-gas than in the original contaminated water stream
- o Provided the air stream is not humid, the competition for adsorption sites for water is lower.

The application of vapor-phase GAC adsorption to the control of stripper off-gases is relatively straightforward. An important difference from solvent recovery applications is that stripper off-gases are saturated with water vapor. The water vapor competes for adsorption sites and effectively reduces the capacity of GAC for organics, so increasing the frequency of regeneration.

In solvent recovery systems, the GAC is regenerated with steam. Effective regeneration can be achieved at relatively low temperatures (down to 150 °C), and is normally done directly in the adsorption unit. This is in

contrast to the regeneration of aqueous-phase GAC, which involves transferring the carbon to a special purpose furnace and heating it by hot combustion gases to high temperatures (up to 1000 °C) (Reference 7). However, the cost of treating the off-gas from an air stripper can be significantly higher than the cost of treating the off-gas from a solvent recovery system because of two factors. Some investigators have reported that relative to solvent recovery, high steam rates are required for regeneration of GAC used for air stripping (Reference 5). In addition, as discussed above, the GAC needs to be regenerated frequently because of the high gas humidity.

Reducing the cost of regeneration is important in developing the vapor-phase adsorption processes for upgrading existing air strippers, and is a necessity if air stripping is to remain a viable treatment option for VOC control for new plants.

One means of decreasing regeneration costs is to reduce the frequency of regeneration by increasing the adsorption capacity of the carbon (References 5, 8 and 9). This can be done by heating the off-gas upstream of the adsorption bed. Heating the off-gases is itself expensive, however, and for low VOC concentrations, can double the operating cost for off-gas control.

The more direct approach is to reduce the cost of the regeneration process itself. Adopting in-place steam generation rather than the high-temperature thermal processes traditionally used in aqueous-phase treatment results in substantial cost savings. Several studies have already shown that regeneration at lower steam temperatures is feasible for desorbing volatile organics (Reference 5).

One method of regenerating GAC at relatively low temperatures is with electrical heating. Although electrical energy is more expensive than thermal energy, the heat is applied directly to the carbon without the need for a carrier gas. This increases the rate of heating without the need for high-temperature gradients, and reduces both heat and carbon losses. Other advantages, such as the reduction in the amount of tail-gas generated and the reduced plant size and complexity might result in reduced overall costs.

### 3. Stripper Off-Gas Heating

Previously, it was pointed out that the adsorptive capacity of the GAC can be increased by heating the off-gas upstream of the adsorption bed. Data presented in the literature (References 5, 8 and 9) show that, especially at low TCE concentrations, the GAC capacity depends on the humidity of the influent air stream, with the driest gas corresponding to the highest capacity. Carbon adsorbs moisture from humid gases which depresses its adsorption capacity for pollutant molecules by as much as a factor of 10 relative to dry carbon. Crittenden (Reference 5) found that increasing the temperature of the influent gas such that its relative humidity drops to about 40 percent is sufficient to restore the carbon capacity to near-normal levels. The added costs to heat the air stripper off-gas prior to adsorption can be as high as 70 percent of the total costs of the untreated case. Additional heating is not beneficial in that the effect of temperature on decreasing the adsorption capacity becomes dominant. Thus, there is an optimum temperature to which the off-gas should be heated to minimize the costs of vapor-phase GAC. This temperature depends largely on the concentration of pollutants in the stripper off-gas (Reference 5) and their vapor pressure-temperature dependence (Reference 8).

Electric resistance and microwave methods provide heat directly to the carbon. The temperature to which the carbon bed is heated can be readily controlled over a wide range simply by regulating the power input. The deleterious effect of humidity on capacity may be counteracted by providing a low level of heat to the GAC during adsorption. The heating costs can be significantly reduced by heating the carbon rather than the large mass of stripper off-gas. When the carbon bed is fully loaded, the adsorption bed is taken off-stream and the carbon is raised to regeneration temperatures by increasing the power input.

Thus, the electric resistance and microwave heating methods may provide both a simple means for in-place regeneration of GAC and a means for overcoming the deleterious effect of humidity on adsorption capacity.

## C. SCOPE

This report is written in 6 sections:

- o Section I - Introduction.
- o Section II - Activated Carbon Regeneration. Current methods of regenerating granular activated carbon are briefly described, together with a discussion of previous work done on the use of Rintoul and microwave heating for vapor-phase and aqueous-phase GAC regeneration.
- o Section III - Experimental Investigation. The components of the laboratory test apparatus, the analytical techniques used for determining the TCE concentrations during adsorption and regeneration, and the procedures for carrying out the test program are detailed. The results of the adsorption and regeneration tests are summarized.
- o Section IV - Cost Analysis. The energy and capital costs of regenerating GAC, humidity control and tail gas treatment by Rintoul heating are estimated and compared to conventional steam regeneration.
- o Section V - Conclusions.
- o Section VI - References.

## SECTION II

### ACTIVATED CARBON REGENERATION

#### A. BACKGROUND

Recent interest in improving the economics of regeneration of activated carbon has triggered commercial development of several new methods. This interest stems from the significant growth in the activated carbon industry in general. For example, the U.S. consumption of activated carbon for vapor-phase processes is projected to increase from 19.8 kilotons in 1987 to 26.8 kilotons by 1992 (Reference 10). The air-stripping process (of which stripper off-gas treatment is a part) discussed here is but one example of many processes where an improved regeneration technique might be applied.

The two principal methods of regenerating vapor-phase GAC are off-site heating at high temperature in a furnace and in-place steam generation. One approach now being promoted is to send the loaded carbon back to the supplier or to a service depot where it is regenerated in a central facility. The carbon is sold in canisters that can be used as the adsorption vessel and which meet United States Department of Transportation standards for shipping. When the carbon is loaded, the canister is simply disconnected and shipped to the central plant for regeneration. This approach is of primary interest to the small user where the capital cost of regeneration equipment cannot be justified.

Although off-site regeneration is not of direct interest here, it does highlight the problem of the small user who needs to minimize capital equipment, for example a boiler for steam regeneration, and a second adsorption vessel to replace the unit undergoing regeneration. A rapid regeneration process that does not incur significant capital costs is sorely needed.

Another major disadvantage of in-situ steam regeneration practiced in the solvent recovery industry is that it produces a polluted water stream that itself has to be treated. To eliminate this, Airco Gases is developing a

process that uses nitrogen in place of the steam (Reference 10). This process is claimed to reduce operating costs by up to 50 percent and has other advantages such as reduced corrosion, reduced carbon degradation, and improved safety.

In another development recently introduced by Radiation Disposal Systems Inc., ozone and ultraviolet light radiation are used to regenerate carbon in a continuous process. This process, which is applicable only to aqueous systems, demonstrates the variety of agents that are used for regeneration.

In general, regeneration processes may be grouped into four classes:

- o Thermal regeneration (References 7 and 11),
- o Extraction with solvents such as methanol (Reference 12 and supercritical fluids such as liquid carbon dioxide (Reference 13),
- o Reaction with chemicals such as carboxylic acids (Reference 14), and oxidation with either air or ozone, and
- o Biological destruction (Reference 15).

Processes that use electrical energy for heat, including infrared systems (Reference 16), can be classed as thermal processes. Steam is also classified as a thermal process rather than as a solvent, and, at high temperatures, can promote reactivation according to the steam-carbon chemical reaction. Ultraviolet light, although generated by electrical input, probably functions more as a catalyst to promote the chemical reaction between ozone and the organics.

In general, all processes require a means of flushing out the desorbed organics and/or regenerant wastes. As stated, the resultant tail-gas is a pollutant stream that itself needs treatment. Processes in which the amount of tail gas produced is both small and easily purified are therefore economically and environmentally attractive.

Incineration is often the purification method of choice, but can be expensive. Recently, however, Allied Signal Engineered Materials have announced new catalysts that destroy volatile organics at lower incineration

temperatures (225 to 335 °C as compared to 490 to 535 °C), and are less sensitive to poisoning by halogens than are conventional catalysts (Reference 17).

The two electrical regeneration methods investigated here are particularly attractive in that the heat is applied directly to the carbon without the need for a carrier gas such as a flue gas or steam. Once the carbon is heated, only a small amount of purge gas may be needed to purge the desorbed organics. These processes are described in more detail below:

#### B. THE RINTOUL PROCESS

The Rintoul process (Reference 18) involves electric resistance heating, where the carbon itself is used as the resistance. The Rintoul process is a proven technology which has been demonstrated extensively in South Africa for carbon-in-pulp plants for the recovery of gold. Several industrial units with capacities of up to 2000 kg are in operation, but for regenerating aqueous-phase GAC in special purpose Rintoul furnaces (Reference 19). The process involves the drying of spent carbon and the use of resistive heating to raise the temperature of the fixed bed of dry carbon to a chosen set point and to hold it constant while superheated steam is passed upwards through the bed to complete the regeneration process. The Rintoul process is used extensively to reactivate heavily contaminated industrial carbon and to further activate or upgrade carbon having low initial activity.

Activated carbon is a semiconductor having a specific electrical resistivity of approximately 5 ohm-cm for fresh dry carbon at an ambient temperature of 20 °C. Regeneration of aqueous phase GAC typically occurs at temperatures in excess of 850 °C. The resistivity of activated carbon decreases by about a factor of seven from 20 °C to 850 °C.

The adaptation to in-place regeneration involves fitting the adsorption bed with electrodes, connecting them to a high-voltage power supply, and providing the necessary electrical insulation. The industrial units were built of

refractory material because they were operated at over 850 °C. This is not necessary for the proposed application.

The advantages of the Rintoul furnace over other furnaces used for high-temperature regeneration processes include high energy efficiency and the use of inert refractories as materials of construction. Also, the Rintoul furnace allows the use of a wide range of temperatures, residence times and steam flow rates through the fixed bed of carbon, all of which make for flexibility of operation.

The improved energy efficiency for the Rintoul process has been presented in Reference 19 and confirmed in a 200 kg pilot unit. These data have been used to estimate energy costs for the proposed application and are shown in Table 1 to be about one-thirtieth of those for steam regeneration. Reasons for this much lower cost are twofold:

- o First, only the carbon is heated and not the 20 times larger amount of steam;
- o Second, heating the carbon involves only sensible heat; the latent heat for generating the steam is not readily recovered.

#### C. MICROWAVE REGENERATION

The regeneration of granular activated carbon by microwave heating has been studied extensively in Germany and Japan during the past 20 years (References 20 to 26). Recently, Fang, et al., (Reference 27) at the University of Southern Louisiana conducted experiments on the microwave regeneration of vapor-phase GAC and aqueous-phase powder activated carbon. The generation of heat by microwaves depends on the presence of dipoles (e.g., water molecules), which, when placed in a rapidly changing electric field, undergo changes in orientation; this can result in "friction" which generates heat. Furthermore, microwave power, with its relatively long wavelength, can pass through a porous layer with little attenuation and be absorbed within the region where contaminants are to be removed. Frequencies available for industrial applications are limited by Federal authorities to two: 915 MHz and 2450 MHz.



TABLE 1. ESTIMATED ENERGY COSTS FOR REGENERATING 1000 KG CARBON  
(Regeneration Temperature = 120 °C; steam @ \$0.011/kg;  
electricity @ \$0.10/kWh)

	Rintoul <u>Furnace</u>	Steam <u>20 kg/kg</u>
Initial heating	\$ 4.95	\$220
Heating purge gas <sup>a</sup>	\$ 0.65	Incl.above
Heat losses (estimated)	\$ 1.00	Incl.above
Drying	<u>b</u>	<u>c</u>
Total	\$ 6.60	\$220.

<sup>a</sup> Based on 0.2 kg gas per kg carbon - See Reference 19.

<sup>b</sup> Not required.

<sup>c</sup> Not calculated.

The following equation gives the amount of power generated in the material in the electric field:

$$\text{Power (W/cm}^3\text{)} = 55 \times 10^{-14} E^2 F e^N$$

where E = electric field strength (V/cm), F = frequency (Hz), and  $e^N$  = loss factor. The loss factor is an intrinsic property of the material which depends strongly on temperature and concentration. In particular, liquid water has a large value of loss factor and thus should couple well with the microwave power. Also, other polar molecules can also efficiently absorb microwave energy.

Theoretically, the use of microwaves should result accelerate the rate of activated carbon regeneration because the heat transfer does not require internal temperature gradients, and the temperature of the activated carbon could be maintained close to the maximum permissible temperature without an associated loss in its adsorptive quality. This is in direct contrast to

conventional radiant and/or conduction heating. In the conventional radiant and/or conduction heating, a temperature gradient is required for the energy to be transferred from the outer porous layer to the inner layer where further regeneration is taking place. A substantial temperature gradient is needed for a reasonable heat transfer rate. To accomplish this, it is necessary to keep the external temperature high. Thus, the external surface of the activated carbon may be exposed to a high temperature which may cause the deterioration of its adsorptive properties. In a cyclic process such as repeated regeneration of porous adsorbent materials, low operating temperatures are beneficial for the prevention of loss of adsorption capacity.

Use of microwave power makes it possible to accelerate the regeneration rate to the diffusional limit of the water vapor through the porous layer without requiring a high-temperature gradient and to achieve an order of magnitude increase in regeneration rate. Other advantages of using microwaves for the regeneration of activated carbons include:

- o Field regeneration of the activated carbon is feasible as it is relatively easy to generate microwaves on a small scale.
- o High-efficiency magnetron coupled with a long tube life makes the use of microwaves for the regeneration of the activated carbon an economical means for such an application.
- o The excellent coupling between microwaves and polar molecules makes the use of energy extremely efficient and thus further reduces the cost of energy for regeneration.
- o An extremely high regeneration rate is possible as the result of volumetric heating of the activated carbon by the penetration of microwave energy.

Microwaves have been used to regenerate activated carbon used for the adsorption of toluene from waste gas (Reference 20). The regeneration of activated carbon by microwaves with steam at high temperature was reported by Yoshida and Tsuru (Reference 21). A double-walled tower with a perforated disk

was used for the adsorption of the contaminants at the top of the tower (400-600 °C), while regeneration of the activated carbon was accomplished with steam injection at the bottom coupled with microwave energy at 2450 and 915 MHz. The use of microwave irradiation for the regeneration of carbonaceous materials from methylene adsorption with steam and with an activated gas was reported by Tanaka, et al. (References 22 and 23). They reported an essentially complete recovery of the adsorption capacity of the regenerated carbon. They further indicated that the use of microwaves is especially useful for the regeneration of spent carbons from wastewater treatment, solvent recovery, and air purification. Similar complete recovery of adsorption capacity by microwave regeneration were also reported by Mashida and Matsushita (Reference 24) and Katsuta (Reference 25) for activated carbons having been exposed in an environment containing methyl acetate, ethyl acetate, methanol and phenol.

The rapid regeneration of spent activated charcoal by microwaves was also demonstrated by Katsuta (Reference 26). The regeneration was done by the irradiation of microwave on free falling charcoal particles in a number of quartz pipes which were sealed in a housing. Again, 100 percent regeneration of the spent charcoal was reported. Fang, et al., (Reference 27) showed that spent activated carbon, generated from the adsorption of n-hexane, acetone or ethanol, was effectively regenerated with microwave radiation without purge gas.

## SECTION III

### EXPERIMENTAL INVESTIGATION

#### A. EXPERIMENTAL EQUIPMENT

##### 1. Selection of GAC and VOC

A GAC and VOC were needed for the test program. The American Water Works Association WATERNET database was searched using the broad terms "carbon" and "regeneration" to determine the GAC and VOC having the most available test data on vapor phase adsorption. Approximately 110 documents were found and all abstracts were stored on a disk file. With the exception of a report by Crittenden, J., et al., (Reference 5), all of the reports were related to aqueous-phase adsorption.

Because of the lack of vapor-phase adsorption data found in WATERNET, a search was conducted of recent publications in the Massachusetts Institute of Technology Library. The search turned up two papers by Fang C-S, et al. (References 27 and 28) on the regeneration of spent activated carbon using microwaves. A number of patents on the microwave regeneration of GAC were also found (References 18 to 26). Also, a number of papers on experimental techniques using TCE and various carbons were found in the literature. These techniques were used to aid in formulating the experimental setup discussed in the next few sections.

The selection of an organic contaminant is also related to its volatility and its ability to be desorbed from GAC at low temperature. For dilute concentrations of many gases and over a fairly wide range for some gases, Henry's Law states that under equilibrium conditions the solubility of gas in a liquid is directly proportional to its partial pressure in the gas phase. The constant of proportionality is known as Henry's Law constant and is dependent on temperature and relatively independent of pressure. The VOCs stripped in an air stripper and entering the activated carbon bed will generally be volatile compounds with a Henry's Law constant greater than

TABLE 2. VOCs THAT CAN BE EASILY REMOVED BY AIR STRIPPING.

<u>VOC</u> <sup>a</sup>	Henry's Law const. <sup>b</sup> <u>(atm)</u>	Normal boil.pt. <u>(°C)</u>	MCL <sup>c</sup> <u>(mg/L)</u>
1,1,1-Trichloroethane <sup>d</sup>	200	74.1	0.2
1,1-Dichloroethylene <sup>d</sup>	9,400		0.007
1,2-Dichloroethane <sup>d</sup>	61	83.7	0.005
Benzene <sup>d</sup>	260	80.1	0.005
Carbon tetrachloride <sup>d</sup>	1,400	76.8	0.005
Chlorobenzene <sup>d</sup>	220	132.1	
Chloroethane	830		
Chloroform	190	61.2	
cis-1,2-Dichloroethylene <sup>d</sup>	9,400		0.07
Dichlorofluoromethane	120,000		
Ethylbenzene	320	136.2	
Methyl bromide	5,200	4.5	
Methyl chloride	440	-24	
Methylene chloride <sup>d</sup>	140	40	
para-Dichlorobenzene <sup>d</sup>		174	0.075
Tetrachloroethylene <sup>d</sup>	1,300	120.8	0.005
Toluene	320	110.8	
Toxaphene	3,500		
trans-1,2-Dichloroethylene <sup>d</sup>	320		0.07
Trichlorobenzene <sup>d</sup>		208.5	
Trichloroethylene <sup>d</sup>	600	87.2	0.005
Trichlorofluoromethane	6,100		
Vinyl chloride <sup>d</sup>	360,000	-12	0.002

<sup>a</sup> See Reference 29 for an extensive list of strippable and non-strippable organics.

<sup>b</sup> Henry's Law Constants are based on a temperature of 20 °C (Reference 1).

<sup>c</sup> Maximum Contaminant Level; values proposed under 1986 amendments to SDWA.

<sup>d</sup> Denotes a VOC regulated under the SDWA.

0.001 atm m<sup>3</sup>/mol (Reference 1). Organic compounds falling in this category are listed in Table 2 together with their boiling points. The 14 VOCs regulated in the 1986 amendments to the Safe Drinking Water Act (SDWA) are included in Table 2.

Previous studies have shown that the adsorption efficiency generally increases with increasing boiling point. Easily volatilized compounds such as those listed in Table 2 are weakly adsorbed and can be desorbed at relatively low temperatures (References 30 and 31). For example, studies have shown that benzene (Reference 30) and the trihalomethanes (THMs) (Reference 31) can be readily stripped from carbon by simply flushing the spent carbon with steam or hot gases. Steaming, at a rate of 5 to 6 kg per kg solute, is also used to regenerate GAC used to control solvent emissions (Reference 6). Also, carbon has been successfully reactivated between 55 and 120 °C with supercritical carbon dioxide (Reference 13).

Calgon BPL granulated activated carbon and trichloroethylene (TCE) were selected for the test program. First, extensive breakthrough and isotherm data are available for the TCE/BPL system (Reference 5). Secondly, TCE is relatively volatile and should give a good indication of the effect of humidity control on improving performance. Chlorinated hydrocarbons are most affected by humidity, while xylenes are least affected.

Table 3 lists some of the relevant physical properties of BPL carbon (Reference 16) and Table 4 shows some of the relevant chemical properties of TCE.

A vapor pressure curve for TCE was used for sizing the experimental equipment. Figure 1 is a calculated vapor pressure curve for TCE. The curve is based on using the vapor pressure versus temperature data from Table 4 to calculate the Antoine coefficients A, B and C (Table 5):

$$\log (\text{vapor pressure, atmospheres}) = A - \{B/[C + T(^{\circ}\text{K})]\}. \quad (1)$$

Table 6 lists selected temperatures and vapor pressures for TCE based on Equation (1), with the corresponding TCE concentration.

TABLE 3. PHYSICAL PROPERTIES OF CALGON BPL CARBON.

Size of carbon (mesh)	4 x 6
Average Particle Diameter (cm)	0.372
Carbon Density (g/cc)	2.1
Apparent Density (g/cc)	0.85
Average Bed Density (g/cc)	0.52-0.55
Bed Void Fraction	0.35-0.39

TABLE 4. CHEMICAL PROPERTIES OF TRICHLOROETHYLENE (TCE).

Molecular Formula	$C_2HCl_3$ ( $CCl_2=CHCl$ )
Molecular Weight	131.4
Boiling Point ( $^{\circ}C$ )	87.2
Vapor Pressure <sup>a</sup>	Temperature
<u>(mm Hg)</u>	<u>(<math>^{\circ}C</math>)</u>
1	-43.8
5	-22.8
10	-12.4
20	-1.0
40	11.9
60	20.0

<sup>a</sup> Reference 32.

TABLE 5. ANTOINE COEFFICIENTS FOR TCE<sup>a</sup>.

<u>Temperature (<math>^{\circ}C</math>)</u>	<u>A</u>	<u>B</u>	<u>C</u>
From -50 to -10	4.78993	1606.72	-19.8887
From -10 to +20	3.29691	956.224	-76.0697

<sup>a</sup>  $\log (\text{vapor pressure, atmospheres}) = A - \{B/[C + T(^{\circ}K)]\}.$

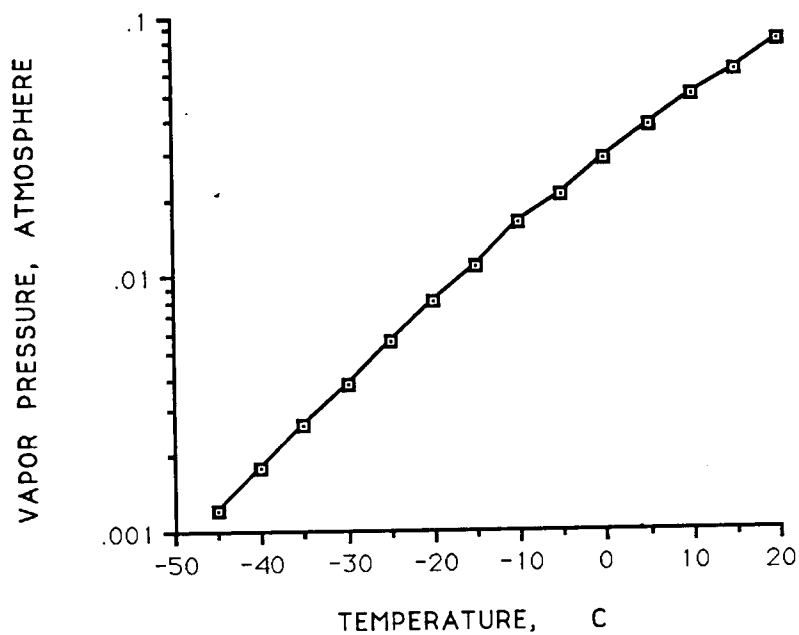


Figure 1. Vapor-Pressure Curve for TCE.

TABLE 6. TCE VAPOR PRESSURE AND CONCENTRATION VS. TEMPERATURE.

Temperature (°C)	Vapor Pressure (atm)	Vapor Concentration (mg/L)
20	0.08	440
10	0.048	272
0	0.028	164
-10	0.015	91

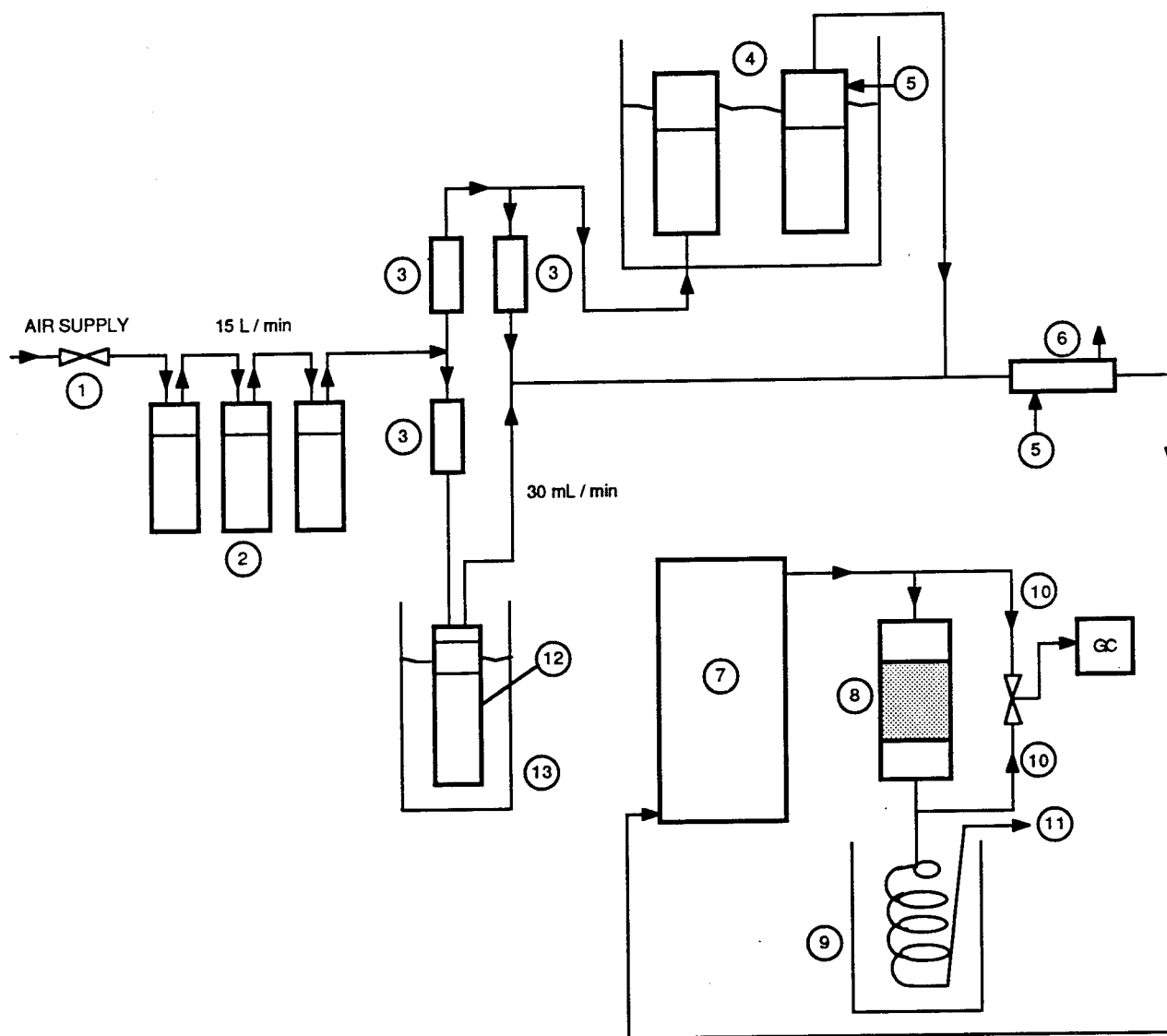


## 2. Flow System

Figure 2 is a schematic of the experimental flow system. This system was designed to obtain information on both loading and regeneration with respect to process repeatability. The flow system is not intended to obtain fundamental data, such as detailed GAC isotherms with TCE, but is intended to show that the carbon bed will maintain its capacity after successive regenerations. On these grounds, it was assumed that variations in temperature and humidity of as much as 10 percent will not significantly affect the results. Variations in the influent TCE concentration are also acceptable, provided the total TCE entering the bed is known to within about 5 percent. The energy requirement for regeneration was the major variable under investigation and was therefore needed to be accurately determined.

The experimental equipment consisted essentially of a vapor-generation system, an adsorption section, and a gas analysis system. The flow system was initially designed to produce an air stream saturated with water vapor and containing TCE at a concentration of about 0.3 mg/L. This is the upper end of the 0.03 to 0.3 mg/L range commonly found in air strippers (Reference 9). However, even at the highest concentration of 0.3 mg/L, the time to breakthrough was estimated to range between 2 to 22 hours, depending on the assumed carbon capacity, which in turn is affected by relative humidity. To obtain many adsorption-regeneration cycles, higher TCE concentrations were generated during the test program. Air streams containing TCE at about 400 mg/L were combined with dilution air to produce an influent TCE concentration of from 4 to 40 mg/L. The higher influent concentrations allowed one adsorption-regeneration cycle to be carried out in 1-2 days.

The concentrated TCE air stream was generated by bubbling dry air through TCE. Depending on the final concentration that was required, the TCE was cooled to as low as  $-25^{\circ}\text{C}$  to reduce its vapor pressure and its concentration. Room air, saturated in a temperature-controlled water bath, was used for dilution.



- |   |                           |
|---|---------------------------|
| 1. TWO-STAGE PRESSURE REGULATOR                                 | 7. EQUALIZATION VESSEL    |
| 2. AIR DRYING (DRIERITE) AND PURIFICATION (GAC)                 | 8. GAC BED                |
| 3. FLOW METER AND NEEDLE VALVE                                  | 9. COLD TRAP              |
| 4. HUMIDIFICATION   | 10. SAMPLING LINES        |
| 5. TAP-WATER SUPPLY   | 11. EFFLUENT TO FUME HOOD |
| 6. TURBULENT MIXING SECTION<br>(WITH WATER JACKET, IF REQUIRED) | 12. TCE COLUMN            |
|   | 13. ICE BATH              |

Figure 2. Schematic Diagram of Experimental Flow System.

a. Vapor Generation

The purpose of this system was to produce a known flow of air containing the desired TCE concentration through the adsorption bed. The linear (empty bed) flow velocity through the carbon column was to be no higher than 50 cm/sec, near the top of the typical operating range of 10 to 51 cm/sec for a carbon column (References 5 and 9). For a nominal one-inch (2.54 cm) inside diameter, the volumetric flow rate should not exceed 15.2 L/min.

b. TCE Supply

Two methods of supplying TCE were considered. In the first a syringe pump was used to inject liquid TCE into an evaporator at the desired rate. For a desired concentration of 0.3 mg/L and a 15 L/min air flow rate, the required TCE injection rate was 4.5 mg/min or about 5 microliters/min.

In the second method and the one used in the test program, the air flow was split and a small bleed stream was diverted through a column filled with TCE. According to Werner, et al., (Reference 8) a bleed stream as low as about 0.2 percent of the total air flow (0.030 L/min) is feasible. To obtain the lowest required TCE concentration of 0.3 mg/L, the concentration of TCE in the bleed stream was maintained at about 150 mg/L, corresponding to a mole fraction of 0.0256 (see Table 6). For a saturated bleed stream, the TCE column was kept just below 0 °C. By decreasing the depth of the impinger-tube below the TCE, a less-than-saturated stream was obtained, making it possible to use higher temperatures. Immersing the TCE column in an ice bath was the most cost-effective approach for obtaining significantly lower TCE concentrations.

If higher TCE concentrations were desired, the temperature of the bath could be increased to room temperature (20 °C), resulting in a TCE concentration of 440 mg/L in the bleed stream (Table 6), or the bleed stream flow rate can also be increased. Influent TCE concentrations of 4 mg/L and 40 mg/L were obtained by operating the bath at room temperature, reducing the primary air flow rate to 7 L/min, and increasing the bleed flow rates to approximately 0.07 L/min and 0.7 L/min, respectively.

#### c. Air Supply and Humidity Control

Since isotherm data were already available, a compressed air supply rather than highly purified bottled air was used. The air supply was dried and the relative humidity was controlled by adding water vapor rather than by heating humid air in a heat exchanger. The air supply was dried by passing it through a desiccant (drierite) as well as precleaned in a large carbon column.

Most of the testing was carried out with air saturated with water vapor. One test run was carried out with dry air. For the saturated case, the entire main air stream was passed through a bubble column filled with water and kept at a fixed temperature (either tap water temperature or at a constant bath temperature). For the case of dry air, all of the air stream bypassed the water column. Condensation in the sampling lines and elsewhere during adsorption was never a problem at room temperature. At a temperature of 20 °C, saturation conditions corresponds to a concentration of 0.0189 g water/liter dry air, or about 1 mL water every 3 minutes for a 15 L/min air flow.

To avoid calibration problems, the air flow rate was measured dry. Because the TCE concentration was measured in the humid air stream, a correction (maximum of 2.3 percent) was applied when calculating the total TCE entering the carbon bed.

#### d. Adsorption Column and Regeneration Apparatus

The adsorption column was a nominal 1-inch (2.54 cm) diameter glass tube with a length of one foot (30.5 cm). This column was able to fit inside the microwave oven. The column was loaded with GAC to a bed depth of 8 inches (20.3 cm).

For Rintoul heating, the carbon was supported between stainless steel screens, which also served as the electrodes. A graphite electrode was not required. In the microwave version, a fritted glass disc supported the carbon bed.

For Rintoul heating, alternating current power was supplied to the stainless steel screens/electrodes from a Variac voltage source. Two multimeters were installed to monitor the power input into the system. The internal temperature of the column was monitored with thermocouples placed uniformly at five locations over the length of the carbon bed; one at the top and bottom, plus another three evenly spaced. The column was insulated to prevent heat loss and also to prevent condensation on the glass walls of the column.

Microwave heating was done using a General Electric Co. Oven Model JE 1465H [height-13.5 inches (34.3 cm), depth-23 inches (58.4 cm), width-14 inches (35.6 cm)] with a maximum power input of 700W. The oven had 10 power settings which provided flexibility to investigate the effects of power input on carbon regeneration.

The microwave oven was modified to accommodate the carbon column and the flow system necessary for TCE analysis. Holes were drilled into the microwave oven and tubing for the flow system was installed. Special silver epoxy (TRA-CON, Inc.) was required to seal the holes created by the flow system and prevent microwave radiation leakage. The microwave oven was tested using a Narda Microline Electromagnetic Radiation Monitor, Model 800, and leakage was found to be less than  $0.05 \text{ mW/cm}^2$ . This value is about one order of magnitude smaller than the OSHA standard for maximum microwave radiation leakage of  $1 \text{ mW/cm}^2$ . The temperature distribution was monitored with a surface pyrometer.

The adsorption column and the regeneration apparatus were placed under an exhaust hood to allow complete dispersion of any (leaky) air containing TCE into the atmosphere.

The effluent gas leaving the carbon column flowed to a cold trap to condense out the TCE prior to discharge.

#### e. Instrumentation

A Perkin-Elmer 8500 Gas Chromatograph (GC) with HS-6 headspace sampler was used to measure the concentration of TCE in the influent and effluent humidified air streams to and from the adsorption column. This system contains a Quadrex column which consists of bonded methyl phenyl cyanopropyl silicone. The flame ionization detector (FID) on the GC is relatively insensitive to water in the humidity range of interest. This column has been used on other programs to detect TCE. A flow system was devised to allow the sampling of TCE from either the influent or the effluent gas stream. The minimum time to obtain a chromatograph for a given sample was 6-7 minutes.

A hydrogen gas detector was obtained to comply with current safety regulations. Hydrogen gas was used in the Flame Ionization Detector on the gas chromatograph for analysis of the TCE.

Two flow meters with high accuracy needle valves (15 L/min full scale) and an extra needle valve for the 30 mL/min bleed stream to the TCE column were used in the TCE vapor generation and air supply systems. An equalization chamber with a capacity of about 30 L was also provided.

A constant temperature bath was used to regulate the water temperature for humidity control. Humidity measurement was not required, provided saturation was attained in the water column and the flow rates of the bypass gas stream were accurately controlled.

Temperatures, currents, voltages, and flow rates were recorded manually as a function of time.

#### B. GAS CHROMATOGRAPH TCE ANALYSIS

Standards for TCE were derived using gas chromatographic headspace analysis. Seven microliters of TCE was dissolved in 50 mL of deionized water giving a TCE concentration of 204 mg/L in water. The mixture was further diluted with deionized water to give TCE concentrations in water of 25, 50 and 100 mg/L. Four mL of each solution was injected into a 10 mL vial and the TCE

concentration in the headspace was analyzed by gas chromatography. The analysis given below relates the TCE concentration in the headspace above the water to the TCE concentration in the water. Trichloroethylene calibration data were also obtained for the vapor generation system and compared to the results obtained by headspace analysis.

The concentration of the TCE in the 6 mL headspace above the water solution was determined from Henry's Law:

$$K = \frac{\text{(mole fraction of TCE in head space)}}{\text{(mole fraction of TCE in liquid)}} = H/P \quad (2)$$

where H = Henry's Law Constant (in atmospheres) and P = total pressure (atm). In the temperature range, T = 15-100 °C, H is given as follows:

$$H = 11T + 375; \quad (3)$$

at room temperature (T = 20 °C), H = 595. Combining Equations (2) and (3), with a total pressure P = 1 atm, the concentration of TCE in the headspace is given by:

$$C_{hs} = 0.45 \times C_{liq} \quad (4)$$

where

$C_{hs}$  = TCE concentration in headspace (mg/L)

$C_{liq}$  = TCE concentration in water (mg/L).

Because of the high volatility of TCE, the TCE in the liquid equilibrates very quickly with the TCE in the gas, and the initial TCE liquid concentration is reduced very rapidly. Using the relationship that the total quantity of TCE in the water initially is equal to the quantity of TCE in the water and headspace after equilibration,

$$C_{tot} (0.4 \text{ mL}) = C_{liq} (0.4 \text{ mL}) + C_{hs} (0.6 \text{ mL}) \quad (5)$$

where  $C_{tot}$  = initial TCE liquid concentration, the TCE concentrations in the headspace and water after equilibration are given as follows:

$$C_{liq} = 0.60 C_{tot} \quad (6)$$

$$C_{hs} = 0.27 C_{tot} \quad (7)$$

Table 7 shows the calculated concentrations of TCE in the liquid and headspace after equilibration as a function of the initial TCE liquid concentration. The total number of moles of TCE found in a 10-microliter sampling volume is shown in the fourth column and is based on the final TCE headspace concentration. The measured peak area for each initial TCE liquid concentration at room temperature and pressure is shown in the last column. The peak area should be directly proportional to the total quantity of TCE sampled by the GC. The data shows that the relationship is not as linear as expected. For example, the ratio of TCE between the highest and lowest values is 8.2 (0.42/0.051), while the ratio of the peak areas is 11.6 (43.6552/3.7665), or a difference of 29 percent.

A solution of pure TCE was also placed in the headspace vial. At room temperature, the calculated vapor pressure of TCE in the headspace is 0.08 atm, which corresponds to a TCE concentration of 440 mg/L (see Figure 1). At the two highest TCE concentrations in Table 7, the average peak area ratio 6.82 (297.62/43.66) correlates with the ratio of the total TCE in the sample 7.98 (3.35/0.42) to within 17 percent.

Calibration measurements were also made in the flow system. Table 8 shows the peak area at a sampling volume of 1 mL and (a) for three flows from the TCE column without any air flow and (b) for air flows of 7 L/min air and 0.7 mL/min TCE at room temperature. The average peak area ratio, 6.46 (17755/2748), does not correlate well with the ratio of the total TCE in the sample, 11.0 (335/30.4). However, there is a fairly good correlation between the pure TCE test in Table 7 and the TCE with air flow test in Table 8, i.e., the peak area ratio is 9.2 (2748/297.62), while the total TCE ratio is 9.1 (30.4/3.35). Another test series was also conducted with similar results.



TABLE 7. TCE GAS CHROMATOGRAPH STANDARDS AT ROOM TEMPERATURE.

Initial Liquid $C_{\text{tot}}$ (mg/L)	<u>TCE Concentration</u>		TCE in GC ( $10^{-8}$ moles) <sup>a</sup>	<u>Peak Area</u>
	Final Liquid $C_{\text{liq}}$ (mg/L)	Final Headspace $C_{\text{hs}}$ (mg/L)		
25	15	6.8	0.051	3.6954 3.8376 <sup>b</sup> 3.7665
50	30	13.5	0.10	7.9780 8.5227 <sup>b</sup> 8.2503
100	60	27.0	0.22	18.3976 18.3788 <sup>b</sup> 18.3882
204	122	55.1	0.42	43.7096 43.6007 <sup>b</sup> 43.6552
Pure TCE		440	3.35	308.0559 287.1845 <sup>b</sup> 297.6202

<sup>a</sup> Injection volume = 10 microliters.

<sup>b</sup> Average value.

TABLE 8. GAS CHROMATOGRAPH ANALYSIS OF FLOW SYSTEM AT ROOM TEMPERATURE.

	TCE Concen. in Gas Stream <u>(mg/L)</u>	TCE in GC <u>(10<sup>-8</sup> moles)<sup>a</sup></u>	<u>Peak Area</u>
Pure TCE	<sup>b</sup> <sub>440</sub>	335	18185
			17519
			17561
			<sup>c</sup> 17755
TCE with Air Flow	<sup>d</sup> <sub>440</sub>	335	17842
TCE with Air Flow	<sup>b</sup> <sub>40</sub>	30.4	2849
			2716
			2678
			<sup>c</sup> 2748
TCE with Air Flow	<sup>d</sup> <sub>40</sub>	30.4	2629

<sup>a</sup> Injection volume = 1 mL.

<sup>b</sup> Test series 1.

<sup>c</sup> Average value.

<sup>d</sup> Test series 2.

During loading of the GAC, the TCE concentrations of the influent and effluent streams were monitored. As mentioned earlier, a flow system was devised to allow sampling and GC analysis of either the influent or the effluent streams. Breakthrough curves, i.e., the ratio of the effluent to influent concentration vs. time, were plotted for each run. The run was terminated when the effluent concentration,  $C$ , equaled the influent concentration,  $C_0$ , i.e. when  $C/C_0 = 1$ . The TCE concentration of the flow during regeneration was also monitored as a function of time.

The TCE concentrations of the influent and effluent streams to and from the adsorption column and the TCE concentration of the effluent stream after regeneration were obtained as follows. First, the TCE concentration of the influent air streams to the adsorption column was in the range of 4 to 40 mg/L. The influent TCE concentration to the adsorption cycle was obtained from peak area GC analysis and correlated with the results shown in the second part of Table 8, namely "TCE with Air Flow":

$$C_o = 40 (P_o/2748) \quad (8)$$

where

$C_o$  = influent TCE concentration (mg/L)

$P_o$  = influent peak area.

The effluent concentration from the adsorption column was normalized with respect to the influent concentration, as follows:

$$C/C_o = P/P_o \quad (9)$$

where

$C$  = effluent TCE concentration in mg/L

$P$  = effluent peak area.

Second, the peak TCE concentration of the effluent stream after regeneration was near the pure TCE results shown in Table 8. The peak TCE concentration during regeneration was correlated as follows:

$$C_p = 440 (P_p/17755) \quad (10)$$

where

$C_p$  = Peak TCE concentration during regeneration (mg/L)

$P_p$  = Highest value of peak area during regeneration.

The TCE concentration during regeneration was normalized with respect to the peak concentration:

$$C_{\text{reg}}/C_p = P_{\text{reg}}/P_p \quad (11)$$

where

$C_{\text{reg}}$  = TCE concentration during regeneration (mg/L)

$P_{\text{reg}}$  = Peak area during regeneration.

## C. RESULTS AND DISCUSSION

### 1. Preliminary Testing

Adsorption testing was started on a carbon column containing 64.7 grams of GAC. The test conditions corresponded to the design conditions of the flow system described in the earlier subsections. The air flow was 15 L/min and was almost fully saturated with water. The relative humidity of the air was measured to be 82 percent. A bleed stream of 30 mL/min of air (0.2 percent of the total air flow) was diverted through a column filled with TCE and immersed in an ice bath. To obtain a concentration of 0.300 mg/L in the saturated air passing through the carbon column, the vapor pressure of TCE in the bleed stream must be 0.0256 atm. If the bleed stream is saturated, the temperature of the TCE bath must be just below 0 °C (see Figure 1 and Table 6). For an influent concentration of 0.3 mg/L and relative humidity of 85 percent, Werner (Reference 33) measured the capacity of GAC to be 0.027 g TCE/g carbon, based on a 50 percent TCE breakthrough. The time to breakthrough was estimated at approximately 6.5 hours. For relatively dry air (5 percent relative humidity) under the same conditions, the capacity of GAC is 0.286 g TCE/g carbon (Reference 33) and the time to breakthrough is 69 hours.

Testing was run over a number of days and breakthrough was not observed even after 19 hours. A leak was detected in the glass tubing going into the equalization vessel, leading to the conclusion that the TCE concentration in air was much less than 0.3 mg/L. All of the glass tubing was replaced with stainless steel.

Further testing was started and air leaks were found in and around the rubber stopper on the equalization vessel. The air leaks were eliminated by reducing the flow rate by one-half, to approximately 7 L/min. Lowering the flow rate reduces the back pressure in the flow system, due to the small diameter tubes leading to the GC from the adsorption column. To maintain the same breakthrough times, the TCE concentration was increased by more than a factor of two by operating the TCE column at room temperature. The vapor pressure of TCE at 20 °C is 0.08 atmospheres, as compared to 0.028 atmospheres at 0 °C (see Figure 1 and Table 6); this represents a 2.9-fold increase in the TCE concentration.

Adsorption testing was started on another carbon column containing 64.7 grams of carbon. Since the prime objective was testing of the two electrical heating methods for regeneration and not loading of the carbon column per se, the influent concentration was increased to reduce the time to breakthrough. However, data by Werner (Reference 33) showed that the GAC capacity also increases with increasing influent concentration. For example, at a relative humidity of 85 percent and an influent concentration of 1.300 mg/L (a factor of 4.3 higher than the influent concentration of 0.3 mg/L), the highest influent concentration tested by Werner, the capacity of GAC was found to be 0.121 g TCE/g carbon, or a factor of 4.5 higher than the value cited earlier for an influent concentration of 0.3 mg/L. For relatively dry air (5 percent relative humidity) the capacity at an influent concentration of 1.3 mg/L was found to be 0.434 g TCE/g carbon, or a factor of 1.5 higher than that for 0.3 mg/L. Therefore, the overall effect of increasing the influent concentration was to reduce the breakthrough time. A total air flow of 7.7 L/min was split and a bleed stream of 0.7 L/min of air was diverted through a column filled with TCE at room temperature. The TCE concentration of the gas entering the carbon column was calculated to be about 40 mg/L, or about 13 times the influent concentration tested earlier. The gas flow was again saturated with water vapor by passing the entire main air stream through a bubble column filled with water and kept at a fixed temperature. Figure 3 shows a breakthrough curve for the TCE and indicates that breakthrough started to occur after 2 hours. The gas flow exiting the carbon column only reached a concentration of 90 percent of that entering the column during a total run time of 4 hours.

The capacity of the carbon for TCE is given by the following equation:

$$\text{Capacity (g TCE/g carbon)} = (UC_o/W) \int_0^{t_b} [1 - (C/C_o)] dt \quad (12)$$

where

U = total air flow (L/min) = 7.7 L/min

C = TCE concentration of gas leaving the carbon column (g/L)

C<sub>o</sub> = TCE concentration of gas entering the carbon column (g/L)  
= 0.040 g/L

W = weight on carbon in column (g) = 64.7 grams

t = time (minutes)

t<sub>b</sub> = time to 100 percent breakthrough (min) = 240 minutes, or

t<sub>b</sub> = time to 10 percent breakthrough (min) = 125 minutes.

The value of the integral, that is, the area between the curve C/C<sub>o</sub> = 1 and the breakthrough curve for 100 percent breakthrough was 145 minutes; for 10 percent breakthrough the area is 124 minutes. The capacity of the carbon for TCE was calculated to be 0.709 grams TCE/g carbon for 100 percent breakthrough and 0.590 grams TCE/g carbon.

Table 9 shows a comparison between the carbon capacities at an influent concentration of 40 mg/L and those of Werner, cited earlier. For a given relative humidity, the GAC capacity levels off with increasing influent concentration. For example, at the higher concentrations, the GAC capacity increases by a factor of 5 (0.62/0.121) for an increase in influent concentration of 13 (40/1.3), while the capacity increase is 4.48 (0.121/0.027) for a concentration increase of 4.3 (1.300/0.300). This is also consistent with the results of Crittenden (Reference 5) and Coutant (Reference 9).

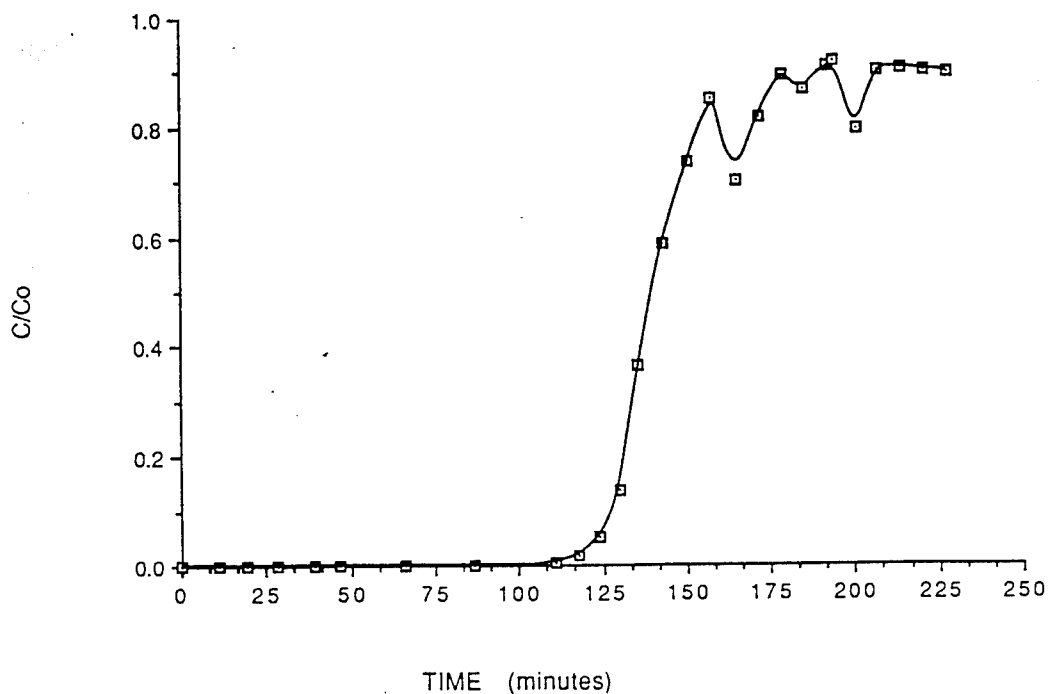


Figure 3. Breakthrough Curve for GAC.

TABLE 9. COMPARISON OF GAC CAPACITIES.

Influent Concentration ( <u>mg/L</u> )	Relative Humidity ( <u>percent</u> )	Capacity (grams TCE/ gram Carbon)	Breakthrough ( <u>percent</u> )
40	82	0.709	100
	82	0.623	50
	82	0.590	10
<sup>a</sup> 1.300	85	0.121	50
	5	0.434	50
<sup>a</sup> 0.300	85	0.027	50
	5	0.286	50

<sup>a</sup> Reference 33.

Regeneration studies of the carbon column were started in a microwave oven. The microwave oven gave nonuniform heating over the length of the column. A surface pyrometer indicated that there was a 50 °C temperature differential between the top and bottom of the column so that not all of the TCE came off and the carbon was not regenerated uniformly. This problem persisted despite the orientation of the column in the microwave or the time spent in the microwave. There was also a considerable problem with arcing in the microwave caused by voltage building up between the grains of carbon. These problems could possibly be controlled with the use of a microwave cavity with a standing wave pattern as opposed to our commercial microwave which has a random wave pattern. Fabrication or purchase of such a cavity was beyond the scope of this program.

Further microwave regeneration testing was not carried out. The remainder of the test program dealt with regeneration by Rintoul heating.

## 2. Adsorption Tests

Ten adsorption tests were carried out on the same GAC sample, which weighed 57.17 grams. A summary of the test program is shown in Table 10. Regeneration of the GAC by Rintoul heating was carried out after each adsorption cycle, except the last. A description of the regeneration tests is described in the next subsection.

The following procedures were carried out on all tests but the first and sixth cycles. The gas flow was humidified by passing the entire main air stream through a bubble column filled with water and maintained at a constant temperature. The relative humidity of the gas was measured at 82 percent. A total (humidified) air flow of 7.7 L/min was split and a bleed stream of 0.7 L/min of air was diverted to a column filled with TCE at room temperature. The TCE concentration of the gas was maintained at approximately 40 mg/L. Once the concentration of the gas flow was established at 40 mg/L in the system, the flow was diverted to the carbon column. As discussed earlier, the influent and effluent concentrations were monitored by a Perkin-Elmer 8500 gas chromatograph equipped with a flame ionization detector and a Quadrex Corporation methyl phenyl cyanopropyl silicone analytical column. The effluent from the carbon



TABLE 10. SUMMARY OF ADSORPTION BREAKTHROUGH DATA.

<u>Cycle</u> <sup>a</sup>	<u>C<sub>o</sub></u> (mg/L)	<u>U</u> (L/min)	<u>Integral</u> 100% (min)	<u>Integral</u> 10% (min)	<u>Capacity</u> 100% (g/g)	<u>Capacity</u> 10% (g/g)	<u>Capacity</u> 50% (g/g)
1	4.2	7.07	908.0	598.0	0.471	0.310	0.413
2	41.1	7.7	120.7	114.7	0.668	0.635	0.713
3	42.1	7.7	152.1	121.8	0.863	0.691	0.778
4	40.7	7.7	162.9	144.5	0.893	0.792	0.869
5	37.6	7.7	172.1	141.0	0.871	0.714	0.817
6	40.9	7.7		168.2		0.926	
7	41.1	7.7		135.9		0.752	
8	37.9	7.7		122.1		0.623	
9	40.1	7.7		118.9		0.642	
<sup>b</sup> 10	40.6	7.6	664.1	624.0	3.588	3.372	3.480

<sup>a</sup> Cycle 6 tested with dry air; other cycles used air with 82 percent relative humidity.

<sup>b</sup> Carbon bed maintained at 50 °C during test.

column was monitored continuously by the gas chromatograph until breakthrough was obtained. Sampling times ranged from 6 to 8 minutes. The concentration of the feed stream was monitored periodically during the breakthrough cycle to ensure its stability.

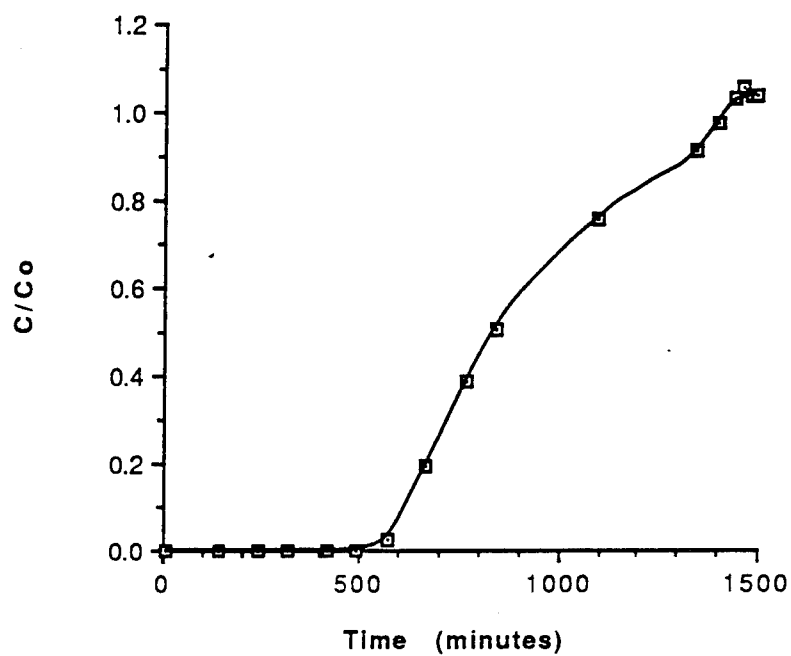
For the first cycle, the total flow was 7.07 L/min and the bleed stream was 0.07 L/min, resulting in an influent TCE concentration of approximately 4 mg/L. For the sixth cycle, the influent TCE concentration was maintained at approximately 40 mg/L, but the air flow was diverted around the bubble column filled with water and was dry.

The first five adsorption tests were run to complete breakthrough ( $C/C_0 = 1$ ), while the last five tests were run to 10 percent breakthrough ( $C/C_0 = 0.1$ ). Figure 4 show the breakthrough curves for all 10 cycles. Table 10 summarizes the results of the adsorption test series. The term "integral" in Columns 4 and 5 refers to the value of the integral in Equation (12), based on 100 percent and 10 percent breakthrough, respectively. The capacity for 50 percent breakthrough is also shown in Table 10.

For 82 percent humidified air and for an influent concentration of approximately 40 mg/L, the 10 percent breakthrough capacity ranges from a high of 0.752 grams TCE/g carbon to a low of 0.623 grams TCE/g carbon, a variation of 27 percent. The average capacity is 0.693 grams TCE/g carbon. The 10 percent breakthrough capacity for an influent concentration of approximately 4 mg/L is 0.310 grams TCE/g carbon (Cycle 1), lower than that for 40 mg/L. For a ten-fold increase in the influent concentration, the breakthrough capacity increases by a factor of 2.2. These trends are consistent with the data of Werner (Reference 33), shown in Table 9. However, at these lower concentrations, for a 4.3 increase in the influent concentration the breakthrough capacity increases by 4.5. Thus, as pointed out earlier, the GAC capacity levels off with increasing influent concentration. Figure 5 shows this trend for 50 percent breakthrough for the data obtained from this study (Table 10) and from Werner (Reference 33).

For dry air with an influent concentration of 40 mg/L, the capacity is 0.926 grams TCE/g carbon. This capacity is higher by a factor 1.34 (0.926/0.623) than the value for 82 percent humidified air. Figure 6 shows the ratio of the dry air capacity to the 82-85 percent humidified air capacity as a function of the influent concentration. The data are taken from the results of the present study (Table 10) and from that of Werner (Table 9). The effect of relative humidity on the GAC capacity decreases with increasing influent concentration.

### RINTOUL COLUMN - BREAKTHROUGH CYCLE 1



### RINTOUL COLUMN - BREAKTHROUGH CYCLE 2

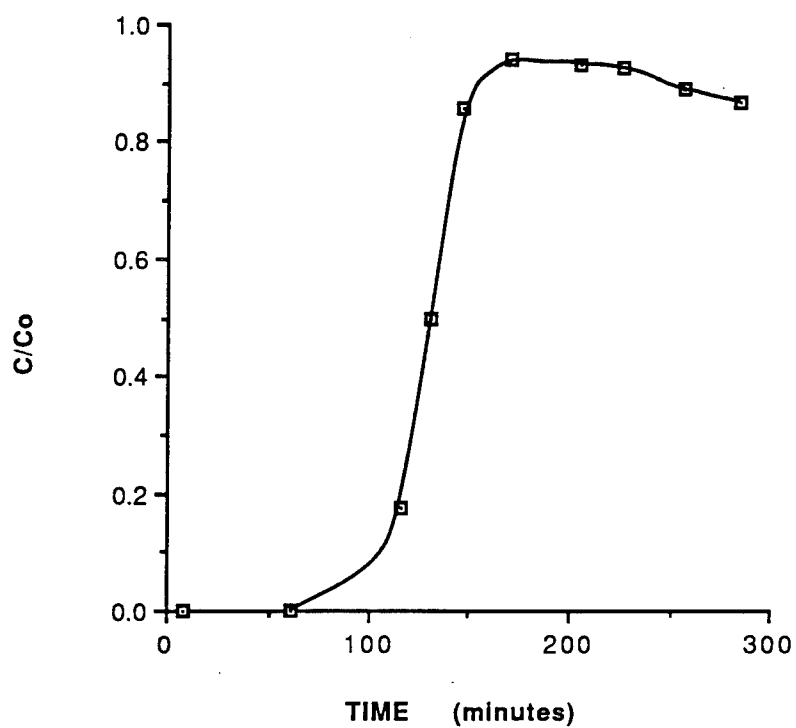
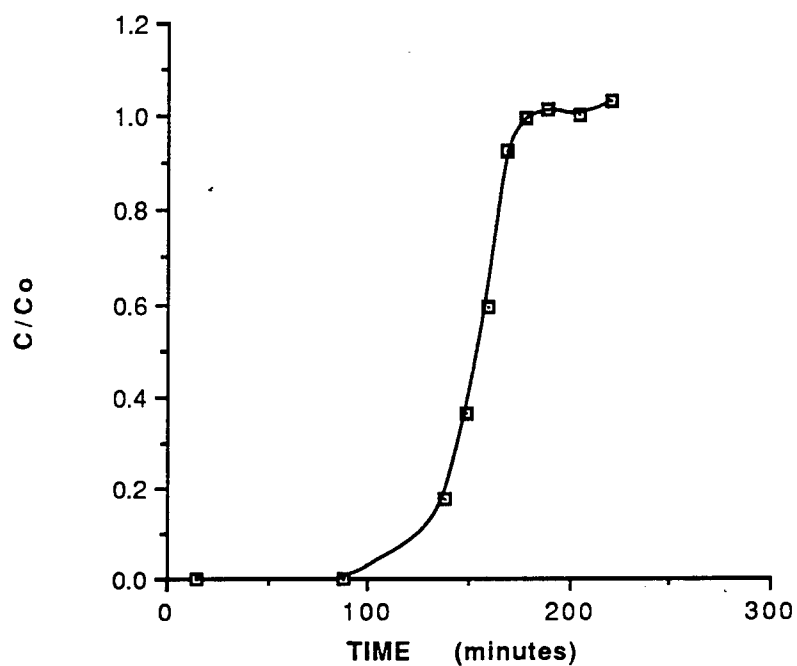


Figure 4. Breakthrough Curves for GAC with Rintoul Regeneration.

### RINTOUL COLUMN - BREAKTHROUGH CYCLE 3



### RINTOUL COLUMN - BREAKTHROUGH CYCLE 4

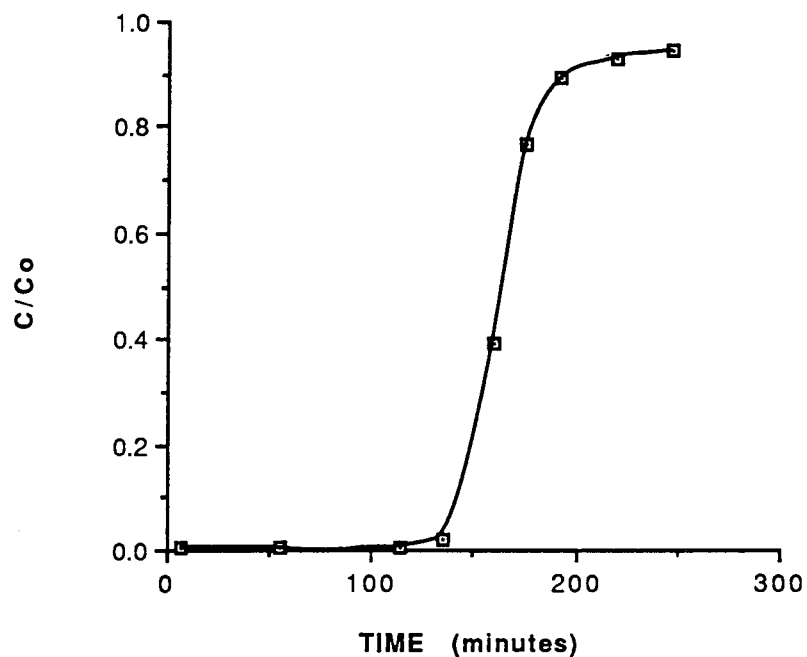
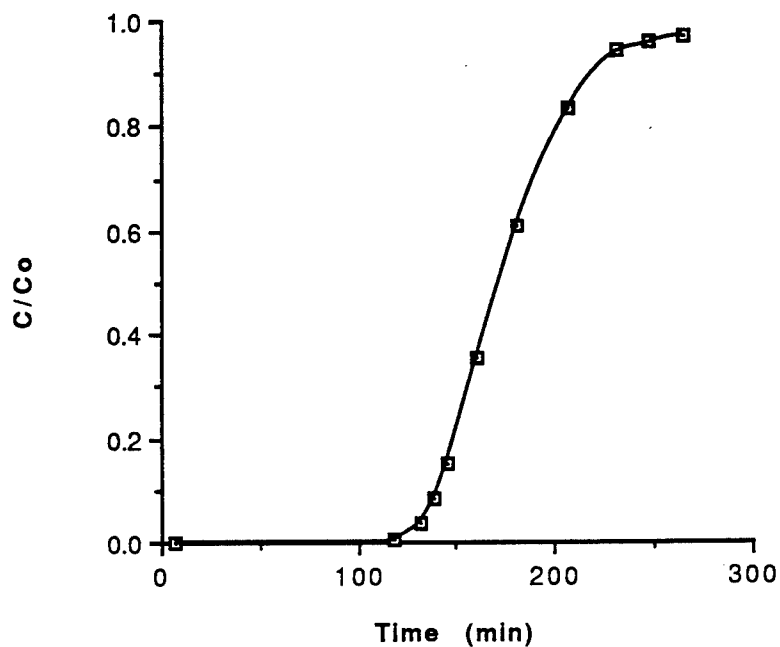


Figure 4. Breakthrough Curves for GAC with Rintoul Regeneration (Continued).

### RINTOUL COLUMN - BREAKTHROUGH CYCLE 5



### RINTOUL COLUMN - BREAKTHROUGH CYCLE 6

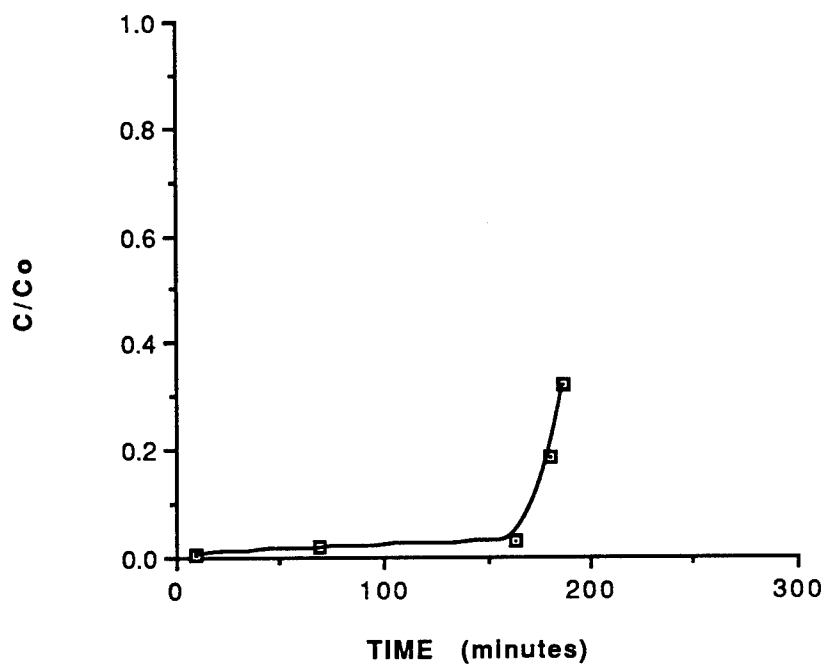
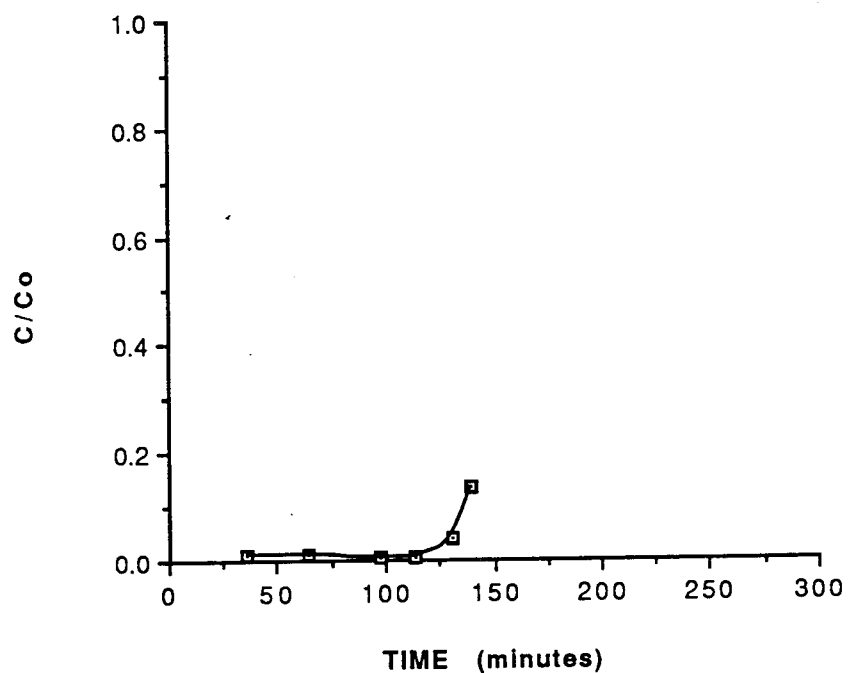


Figure 4. Breakthrough Curves for GAC with Rintoul Regeneration (Continued).

### RINTOUL COLUMN - BREAKTHROUGH CYCLE 7



### RINTOUL COLUMN - BREAKTHROUGH CYCLE 8

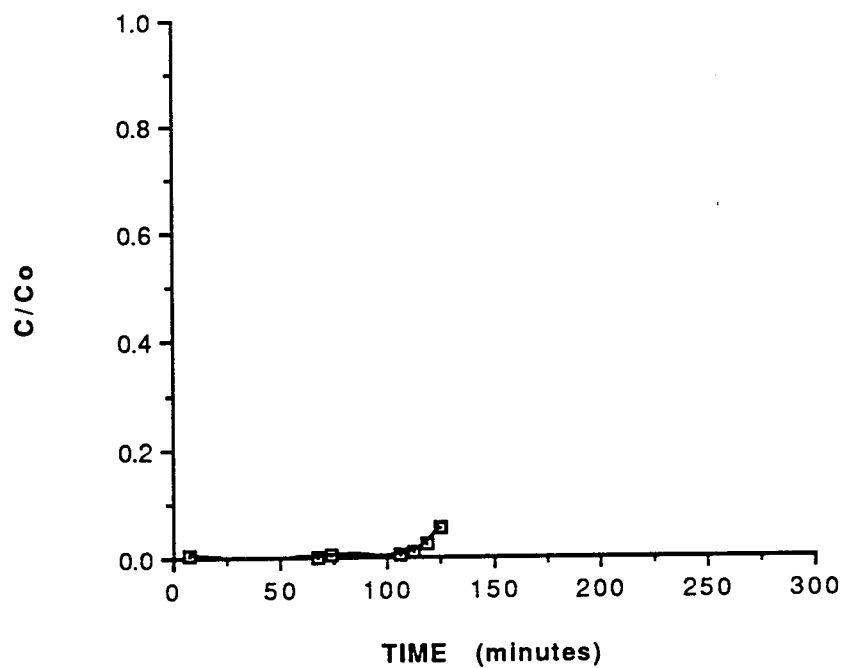
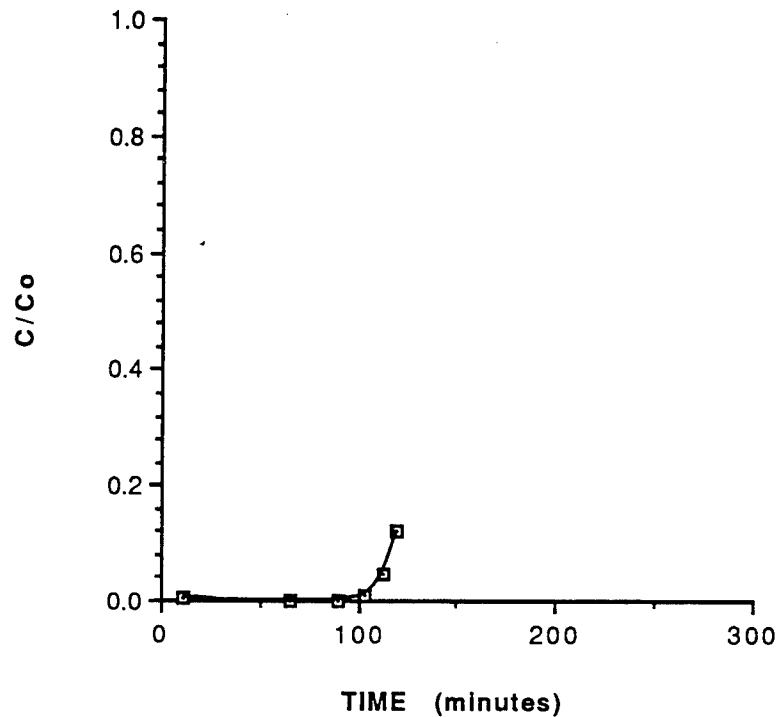


Figure 4. Breakthrough Curves for GAC with Rintoul Regeneration (Continued).

### RINTOUL COLUMN - BREAKTHROUGH CYCLE 9



### RINTOUL COLUMN - BREAKTHROUGH CYCLE 10

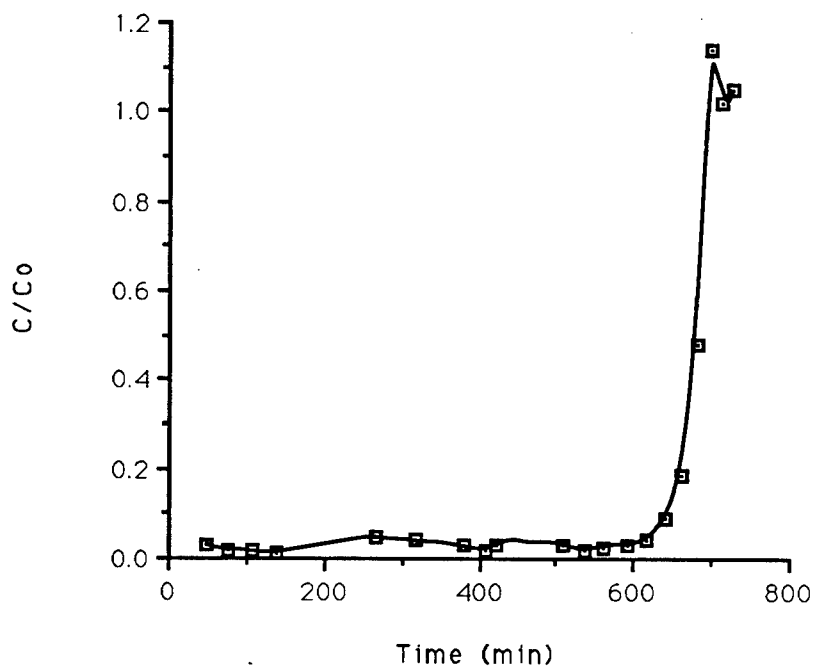


Figure 4. Breakthrough Curves for GAC with Rintoul Regeneration (Concluded).

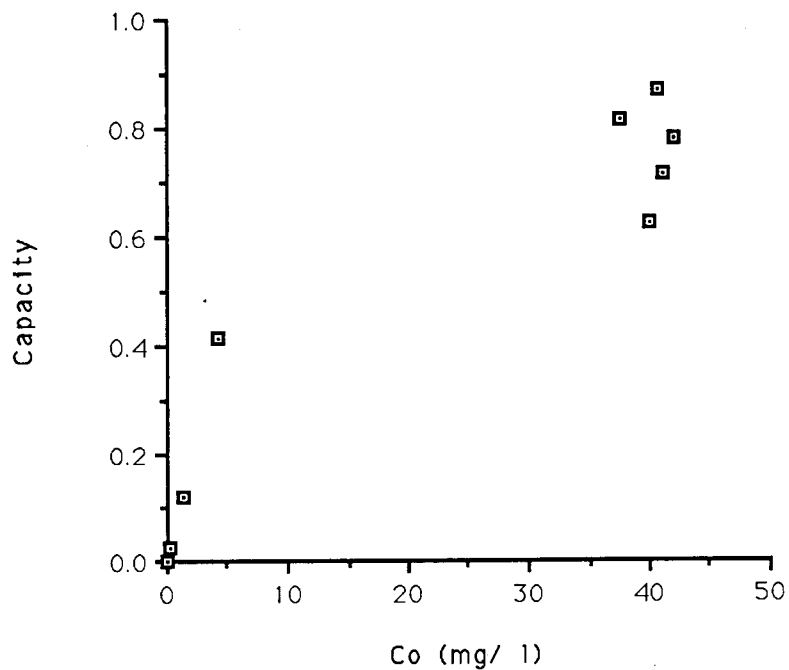


Figure 5. Variation of GAC Capacity with Influent TCE Concentration.

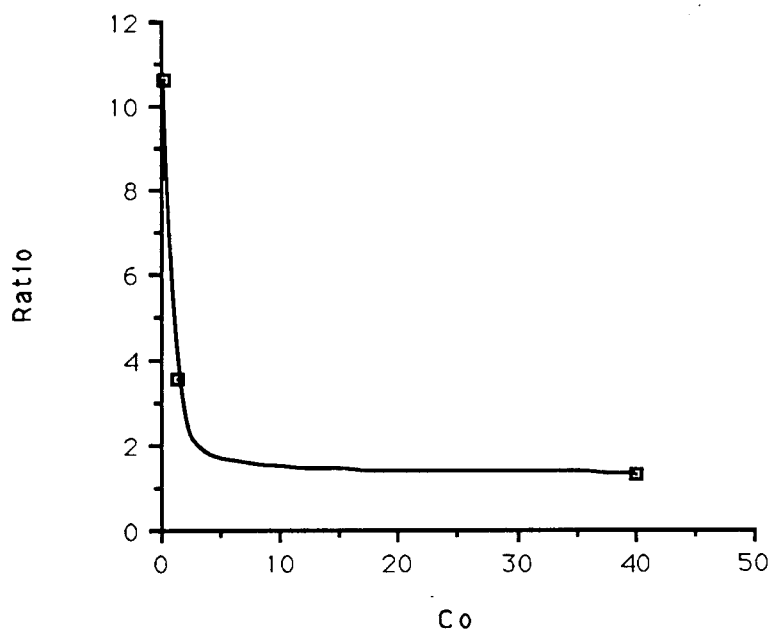


Figure 6. Ratio of Dry Air to Humidified Air Capacities vs. Influent TCE Concentration.



The variation in the capacity of the regenerated carbon bed over 8 cycles is shown in Figure 7. With the exception of Cycle 6 which was loaded using dry air, the influent air stream was humidified to a relative humidity of 82 percent. Although there is a variation of 27 percent in the capacity data for the humidified air, there is no definite trend to indicate a loss of capacity with increasing cycle number. It was concluded that regeneration was accomplished rapidly and effectively and there is little irreversible loss in activity when regenerating with Rintoul heating.

The last test was carried out to determine if low-level heating of the GAC during adsorption would counteract the deleterious effect of humidity on TCE capacity. At high relative humidities, capillary condensation takes place in the smaller pores of the GAC and hinders the adsorption of the VOCs (Reference 5). An increase in temperature increases the pressure at which condensation occurs and so, for a given humidity, decreases the pore size in which condensation can occur. Heating the carbon will therefore decrease the surface area where water can condense. Because the carbon is heated directly, this process should consume less energy than heating the off-gas. The energy saved depends on the temperature to which the bed must be heated and the rate of heat transfer from the bed to the gas. For example, if the off-gas is saturated at 10 °C, heating it to 24 °C will reduce its relative humidity to 40 percent, virtually eliminating condensation (Reference 5). However, the dependence on vapor pressure on temperature is greater for water than for many VOCs. Therefore, increasing the bed temperature reduces the water capacity faster than the VOC capacity and the net effect is a gain in the VOC capacity relative to the unheated case (Reference 9). This has been demonstrated in the laboratory for several VOCs (Reference 8).

During the final test (cycle 10 in Figure 4 and Table 10) the carbon bed was heated to approximately 50 °C and maintained at that temperature (to within 3 °C) during the run. The operating condition of the influent feed air rate was the same as in previous runs, namely, the flow rate was 7.6 L/min, the TCE concentration was approximately 40 mg/L, and the relative humidity was 82 percent. The effluent TCE concentration leveled off after initial startup and ranged from about 1 to 3 percent of the influent concentration. This value was higher by about a factor of 10 to 100 than the effluent concentration before

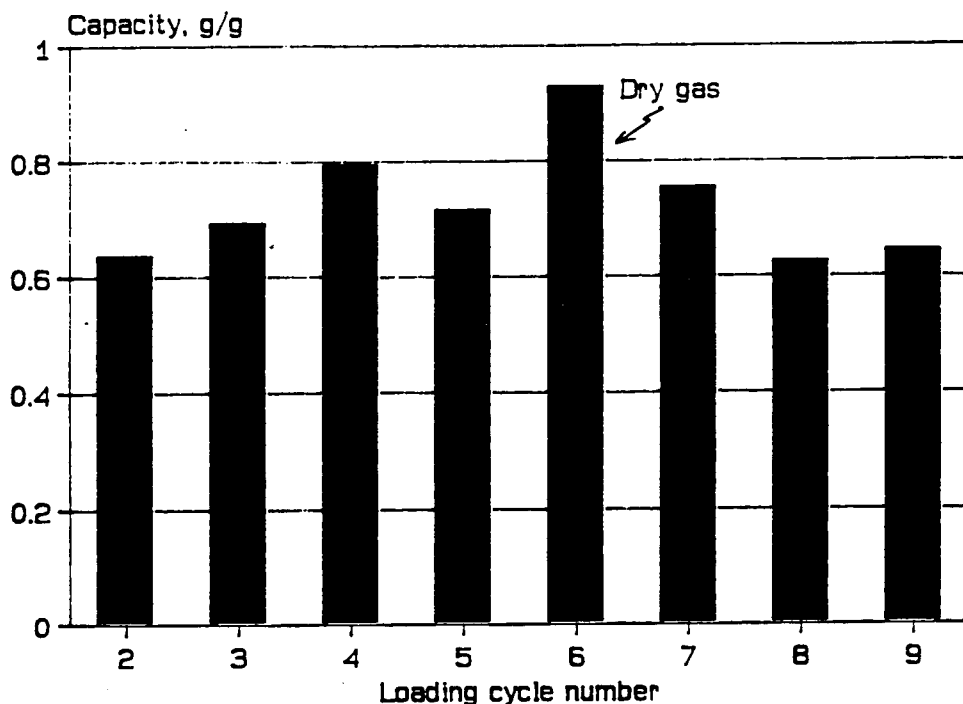


Figure 7. Capacity of GAC Regenerated by Rintoul Heating.

breakthrough of the nine other cycles. Breakthrough (10 percent) of TCE occurred at about 10 hours, or a factor of five greater than for most of the cycles. The capacity also increased by a factor of five over the capacities for 82 percent humidified air, indicating that the capacity of GAC for water decreased faster than the TCE capacity, as discussed above. The average power consumed was 4.29 W over the length of the test run. Thus, during adsorption, direct low-level heating of the carbon bed increased the adsorption capacity at the expense of increased energy consumption. Optimization studies should be carried out to determine whether the overall energy consumed during both adsorption and regeneration can be reduced below the value for heating only during regeneration.

Thus, the following conclusions can be drawn from the adsorption test data:

- o Clearly, the test data obtained for vapor-phase GAC capacity at high influent TCE concentrations with regeneration by Rintoul heating is consistent with other published data at lower TCE concentrations with more conventional methods of regeneration.
- o Similar results are expected to be obtained at lower concentrations with regeneration by Rintoul heating.
- o Furthermore, one of the main objective of the program has been satisfied, namely that the GAC will maintain its capacity after successive regenerations with Rintoul heating.
- o During adsorption, low level heating of the GAC results in an increased carbon column capacity at the expense of increased energy consumption.

### 3. Regeneration Tests

After loading the carbon bed with TCE, the GAC was regenerated by electrical resistance heating, using the carbon itself as the resistance. As described earlier, alternating current power was supplied to stainless steel electrodes (which also served as supports for the GAC) from a Variac voltage source. Generally, the Variac setting was kept constant during the regeneration cycle. The power input to the electrodes, as well as the GAC temperature, was monitored throughout the regeneration run. After the GAC reached the proper temperature, a dry air stream (0 percent humidity) was used to sweep the column. This effluent stream was monitored continuously by the gas chromatograph for TCE concentration.

During each regeneration cycle, the column was heated to approximately 125-180 °C, as measured by the thermocouple at the center of the column. The temperature differential over the length of the column was 20 °C. Air flow through the column was started when the column reached approximately 50 °C and was terminated when the TCE concentration declined. The flow rate for the first cycle was 0.4 L/min, but the results were inconclusive, as discussed below. The air sweep flow rate for most of the cycles was 3 L/min.

Regeneration experiments were also performed at 6 L/min and 15 L/min air flow in an attempt to shorten the regeneration cycle. The 6 L/min experiment was successful but the temperature was difficult to maintain at 15 L/min. Table 11 summarizes the regeneration tests; the regeneration data are shown in Figure 8.

The quantity of TCE released during regeneration was determined by the following equation:

TCE Regeneration Capacity (g TCE/g carbon) =

$$(VC_o/W) \int_0^{t_{\text{reg}}} (C_{\text{reg}}/C_o) dt \quad (13)$$

where

V = air sweep flow (L/min)

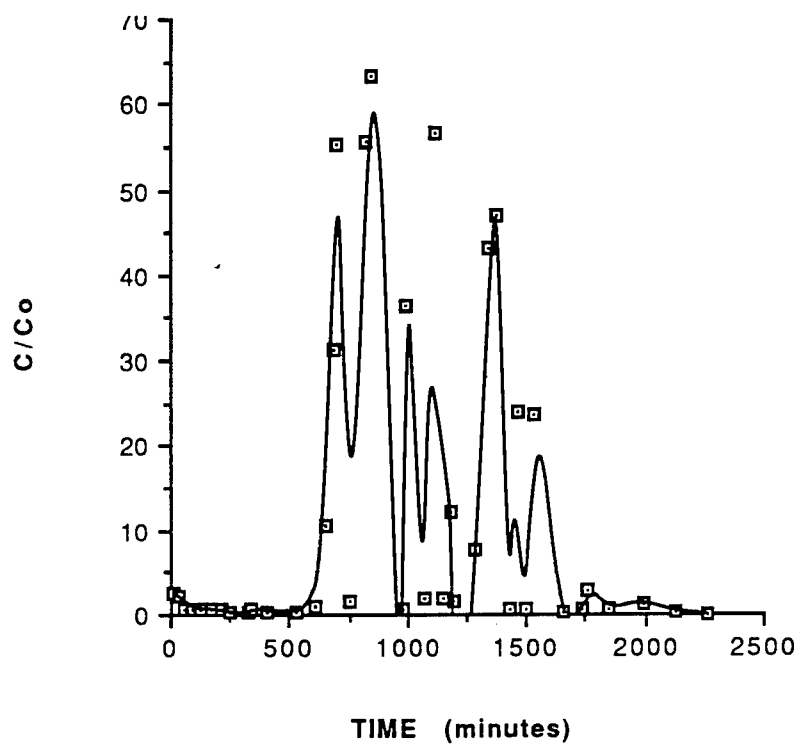
C<sub>reg</sub> = TCE regeneration concentration (g/L)

t<sub>reg</sub> = time to complete regeneration (min);

the remainder of the quantities were defined earlier in this section. The term "integral" in column 4 of Table 11 refers to the value of the integral in Equation (13).

Several problems were encountered during regeneration by Rintoul heating. One major problem was condensation on the internal walls of the column as it was being heated and led to the large fluctuations in the effluent concentration (see Figure 8 - Cycle 1). This was solved by insulating the column after the first cycle and by using a dry (0 °C humidity) air stream for the regeneration cycle. Insulating the column also reduced the heat loss through the walls of the glass column and increased the uniformity in temperature of the column. The column tended to be the hottest near the top electrode and coolest at the bottom with approximately a 20 °C temperature

## RINTOUL COLUMN - REGENERATION CYCLE 1



## RINTOUL COLUMN - REGENERATION CYCLE 2

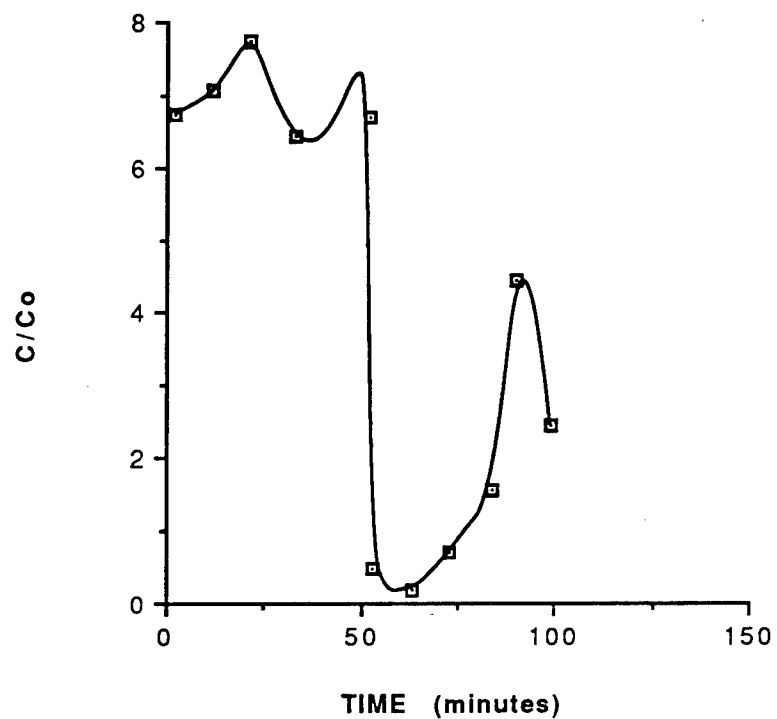
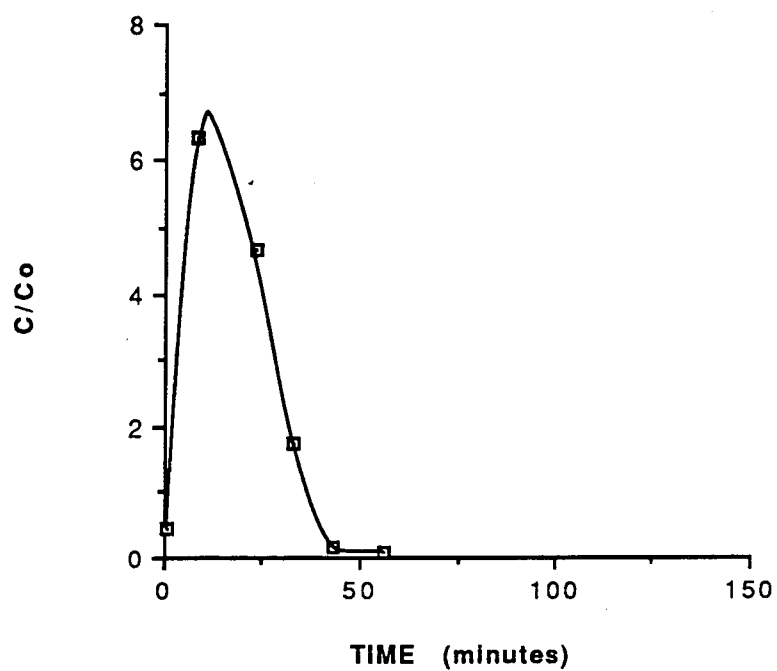


Figure 8. TCE Concentration During Regeneration.

### RINTOUL COLUMN - REGENERATION CYCLE 3



### RINTOUL COLUMN - REGENERATION CYCLE 4

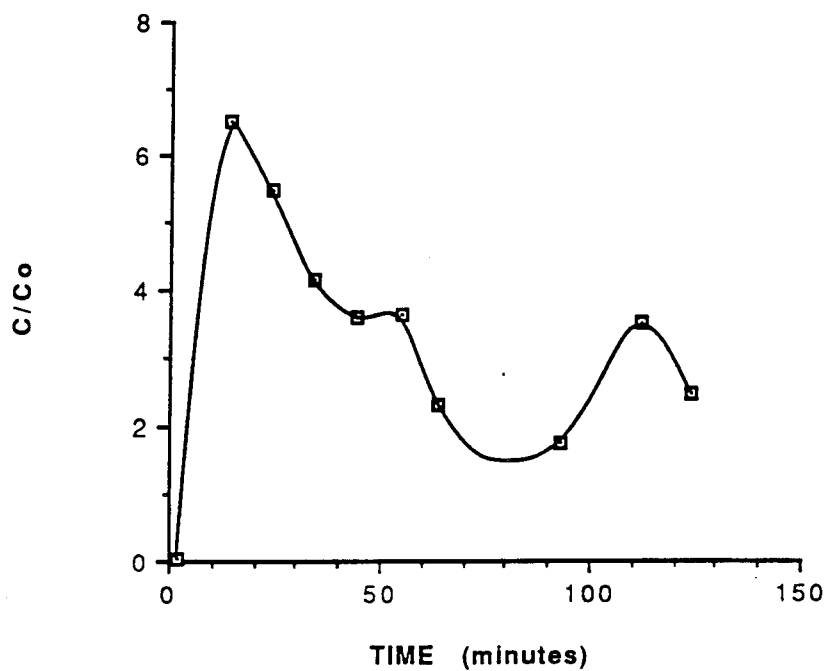
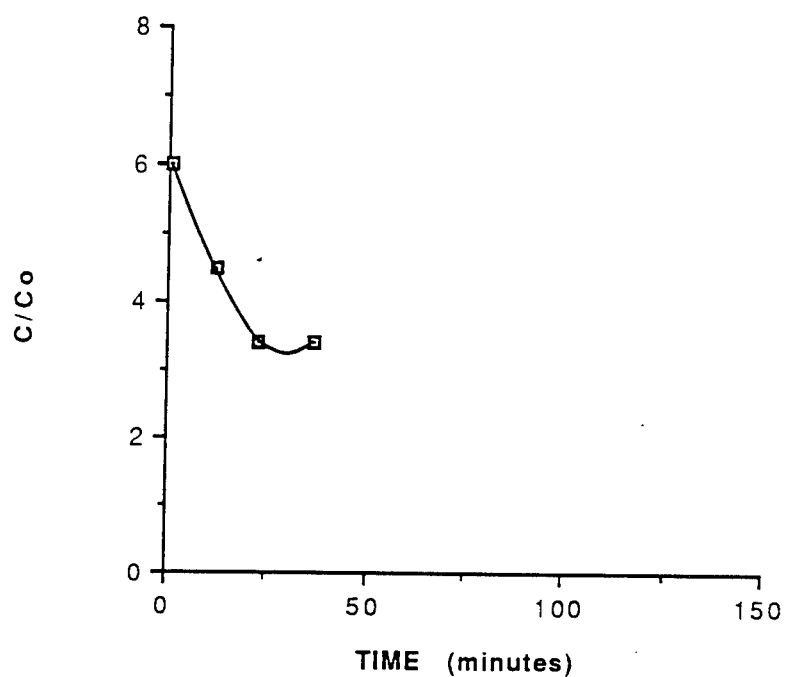


Figure 8. TCE Concentration During Regeneration (Continued).

### RINTOUL COLUMN - REGENERATION CYCLE 5



### RINTOUL COLUMN - REGENERATION CYCLE 6

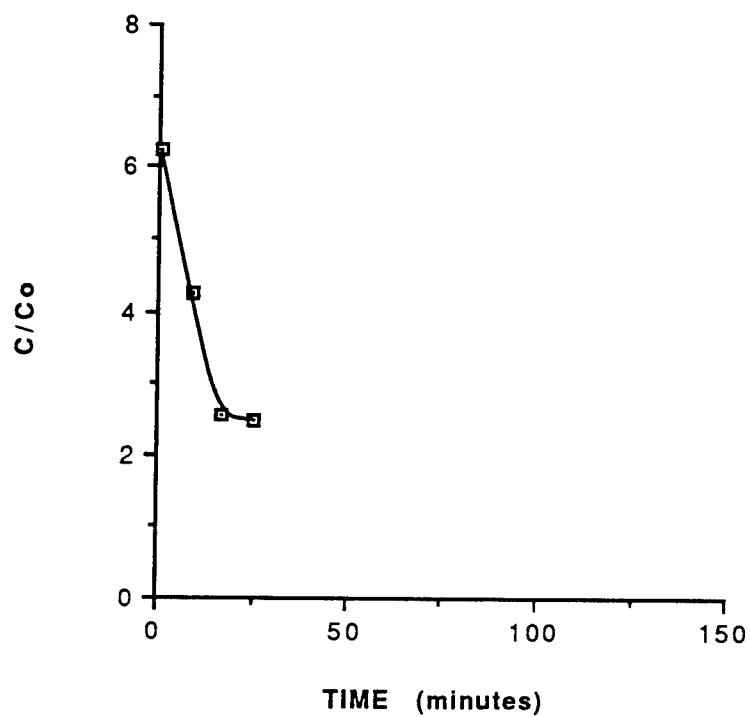
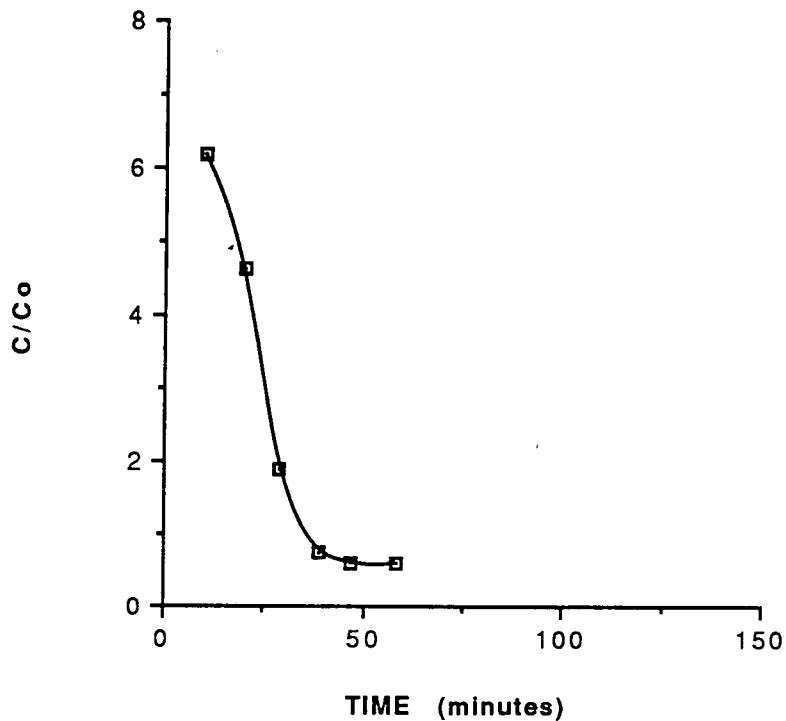


Figure 8. TCE Concentration During Regeneration (Continued).

### RINTOUL COLUMN - REGENERATION CYCLE 7



### RINTOUL COLUMN - REGENERATION CYCLE 8

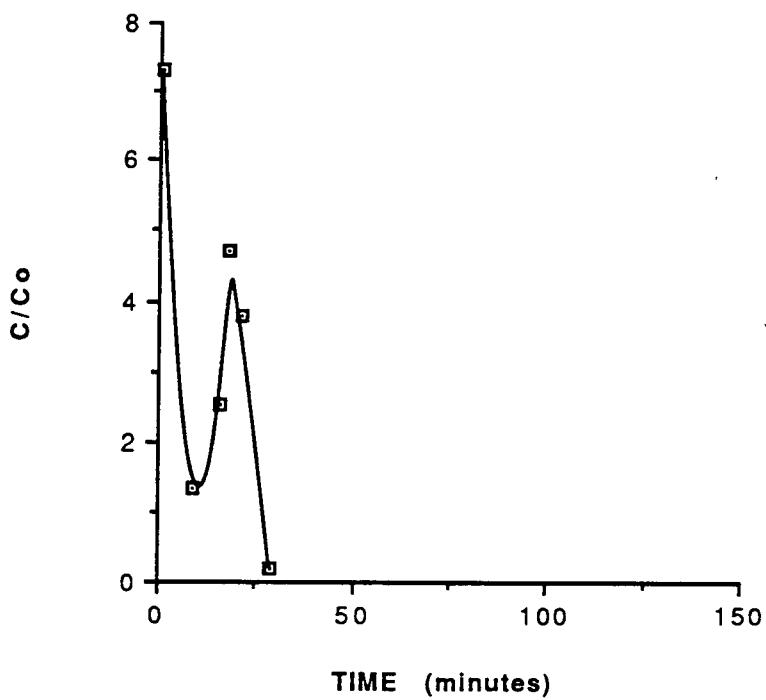


Figure 8. TCE Concentration During Regeneration (Concluded).



TABLE 11. SUMMARY OF RINTOUL HEATING REGENERATION DATA.

<u>Cycle</u>	<u>C<sub>o</sub></u> (mg/L)	<u>V</u> (L/min)	<u>Integral</u> (min)	<u>Capacity</u> (g TCE/g carbon)
1	4.2	0.4	22252	0.654
2	41.1	3.0	425	0.916
3.	42.1	3.0	155	0.343
4	40.7	3.0	396	0.846
5	37.6	3.0	68.0	0.134
<sup>a</sup> 6	40.9	3.0	56.8	0.122
7	41.1	6.0	109	0.470
8	37.9	15.0	73.9	0.735
9	40.1	3.0-6.0	<sup>b</sup>	<sup>b</sup>

<sup>a</sup> New electrodes placed on column.

<sup>b</sup> Not measured.

differential over the length of the column after the insulation was added. There was a 50 °C temperature differential before the insulation was added. During the regeneration cycle, the carbon bed mid-point temperature was recorded for each sample injected into the gas chromatograph.

Another troublesome problem was the temperature fluctuations caused by poor contact between the carbon particles in the carbon bed. This was partially overcome by carefully repacking the carbon bed and vibrating the glass column. Electrode corrosion problems also contributed to temperature fluctuations and led to the replacement of both electrodes between Cycles 5 and 6. Regeneration Cycle 5 was terminated early because of extensive arcing around the bottom electrode and power readings indicated that the bottom electrode was not working properly. The carbon bed temperature was also more difficult to control as the air flow increased from 3 L/min to 15 L/min.

A comparison of Table 10 with Table 11 shows that even after an apparently poor regeneration (Cycles 3, 5 and 6), good adsorption capacities were achieved (Cycles 4, 6 and 7). The measured regeneration capacities are inaccurate because (a) regeneration sampling was done every 8-12 minutes so that it is possible that the top of the elution peak may have been missed and (b) condensation is occurring in the sampling lines, reducing the total TCE detected by the gas chromatograph. There is substantial evidence that the latter is the main reason for the difference between the adsorption and regeneration capacities.

- o First, the same line used for sampling the effluent gas from the adsorption column was used for sampling the purge gas during regeneration; a different line was used for sampling the feed concentration. If the purge gas condenses in the sampling line during regeneration, some condensate should be found in the line during the next adsorption cycle. This was indeed found to be the case. On all of the adsorption runs, but the first, the initial TCE concentrations were all higher than the concentrations measured after a few minutes of testing, indicating residual TCE in the sampling line. In fact, some of the initial concentrations ranged as high 40 to 120 mg/L. However, because the effluent gas is relatively dry (see discussion below), the condensate from the previous run evaporated quickly. The TCE concentrations leveled off at less than 0.1 percent of the influent concentration within a few minutes after startup, and remained at that value until breakthrough started to occur.
- o Second, as discussed earlier, condensation was observed on the internal walls of the glass column during Cycle 1 regeneration and was indirectly due to water adsorption by the GAC during the adsorption cycle. Desorption of the water during regeneration resulted in supersaturation of the purge gas. Since the sampling lines were not insulated and/or heated, condensation would also be expected in the lines. Condensation in the effluent sampling lines during adsorption did not occur because (a) the influent gas flow was not saturated (82 percent relative humidity) and (b) the GAC adsorbed water so the

effluent gas would be relatively dry. Humidity measurements were not made during the adsorption cycle.

- o Third, after the last regeneration cycle, the sampling lines were taken apart and inspected. That portion of the effluent/purge gas sampling line made out of stainless steel was corroded, again indicating condensation had occurred during regeneration. It is important to remember this when designing a larger installation.

On the other hand, we are unable to explain why sporadic condensation occurred, namely, the calculated regeneration capacities were lower on some runs and higher on others. In any event, complete regeneration was always achieved as measured by the adsorption breakthrough capacity.

The temperature of the carbon bed was monitored during regeneration. The higher the carbon bed temperature, the higher should be the rate of desorption and the higher the heat loss to the purge gas. Because the heat loss to the purge gas was relatively small (see discussion below), the carbon should be made as hot as possible. However, the temperature is constrained to the value at which the carbon starts oxidizing (burning) in air. Table 12 shows the maximum and minimum temperatures measured at the center of the carbon bed for each cycle. The maximum temperature ranged from a high of 188 °C to a low of 126 °C. Because the regeneration capacity data is suspect, the optimum desorption temperature and the rate of desorption could not be determined. However, complete desorption must have taken place over the temperature range because the adsorption capacity of the GAC was maintained after each regeneration.

Table 13 shows the power consumed as a function of time and the total energy consumed for each cycle. The Variac voltage setting was generally kept constant during the regeneration cycle. The variations in the input power were a result of the change in resistivity of the carbon bed. The power measurements were only made for Cycles 4 through 9. The energy consumed ranged from a high of 33.0 watt-hours (Wh) for cycle 4 to a low of 4.4 Wh for Cycle 5. However, the high energy consumption of Cycle 4 is a result of the long regeneration time, namely 2 hours, while regeneration Cycle 5 was

TABLE 12. MEASURED TEMPERATURES IN CARBON BED DURING REGENERATION.

<u>Cycle</u>	Maximum Temperature (°C)	Minimum Temperature (°C)
1	188	48
2	155	34
3	126	62
4	159	37
5	159	78
6	177	96
7	127	24
8	177	27
9	177	81

terminated early because the bottom electrode was not working properly. Therefore, a more realistic energy consumption range would be 8.2 Wh (Cycle 6) to 12.4 Wh (Cycle 7); the average power ranges from 16.4 W (Cycle 6) to 48.0 W (Cycle 8). The higher power results in a higher maximum temperature of 177 °C with a relatively short regeneration time (18 minutes). For purposes of cost estimation, the energy consumption for regeneration will be taken as 0.213 kWh/kg carbon (12.2 watt-hours/57.2 g carbon).

Another important consideration is the amount of energy that is absorbed by the gas flow through the carbon bed during regeneration (Table 14). At the lowest flow rate, the temperature of the air stream increases by only 2 °C (22 °C - 20 °C) over the entire carbon bed temperature range. At the highest flow rate, the temperature of the air stream increased by 16 °C relative to a 129 °C increase in the temperature of the carbon bed. The energy adsorbed by the air stream was small compared to the energy increase by the carbon bed. The time it took to reach a given carbon temperature also increased as the flow rate was increased.

TABLE 13. POWER INPUT TO CARBON BED DURING REGENERATION.

Cycle 4	Energy = 33.0 Wh						
Time (min)	2	14	24	34	44	55	64
Power (W)	54.3	21.9	9.2	0.0	16.7	15.2	15.9
Time (min)	93	112	124	125			
Power (W)	15.5	10.8	10.8	off			
Cycle 5	Energy = 4.4 Wh						
Time (min)	1	12	23	37	45		
Power (W)	23.7	6.9	7.9	0.0	off		
Cycle 6	Energy = 8.2 Wh						
Time (min)	1	9	17	25	26		
Power (W)	29.7	26.4	7.5	18.3	off		
Cycle 7	Energy = 12.4 Wh						
Time (min)	0	10	20	29	39	40	
Power (W)	51.0	19.6	9.9	38.7	0	off	
Cycle 8	Energy = 12.2 Wh						
Time (min)	1	9	16	18	21	29	
Power (W)	19.2	61.8	51.4	49.3	0.0	off	

#### 4. Condensate Collection

One of the objectives of the program was to select and test a process that would enable the recovery of TCE given off during GAC regeneration by Rintoul heating. Two tests were carried out. In the first test, the walls of the glass column were not insulated (Cycle 1). During regeneration of the carbon bed, condensate formed on the internal walls of the glass column and drained towards the bottom of the column. The condensate was collected in a

TABLE 14. CARBON BED TEMPERATURES VS. AIR TEMPERATURES.

Flow Rate (L/min)	Temperature ( $^{\circ}$ C)	
	<u>Carbon Bed</u>	<u>Air Stream</u>
3	31	22
	59	22
	93	23
	116	22
	146	22
	154	22
6	25	25
	38	25
	49	27
	57	34
	93	35
	149	36

tube after the power was terminated. For a large-scale operation, the condensate would be collected in the following manner. The carbon would be enclosed in a cylindrical screen. The cylinder would then be enclosed within a water-cooled cylinder. During regeneration, the TCE would be desorbed and condensate containing the TCE would drain down the outer walls of the cylinder and would be collected in an underflow drain system.

The second test was carried out after the final breakthrough cycle (Number 9) was completed. An experiment was performed to collect the TCE that had been adsorbed onto the carbon column. The carbon column was disconnected from the gas chromatograph and connected to a collection vessel that was placed in an ice bath at  $-2^{\circ}$ C. The insulation was also removed from the carbon column to allow the glass to cool. The column was heated (at a power of 90.2 watts) to  $80^{\circ}$ C and condensation began to form on the walls of the column. A humid air stream was then introduced into the column at 6 L/min for

approximately 20 seconds. The condensation dried out immediately and did not reform as long as the air was flowing. As the column continued heating, the condensation reformed at 100 °C and began to flow down the column. A 10-second burst of air was introduced at this point, generating a tremendous amount of sparking in the column. When the column reached 175 °C, the power was shut off and the air supply was again introduced at 3 L/min. The temperature held constant for 5-6 minutes during which time the condensate was removed from the column by the air stream. The condensate was then collected in a 40 mL serum vial. A total volume of 8 mL was collected with 1.5 mL of that being TCE; the TCE was found on top of the condensate. A 10-microliter headspace sample was drawn from the serum vial and injected directly into the Perkin-Elmer gas chromatograph for analysis of the TCE. As expected, the TCE concentration was near that of the solution of pure TCE, shown in Table 7.

Thus, a second method of collecting the TCE is to pass the heated air into a liquid trap at low temperatures. Both methods of collecting condensate containing TCE will be further examined in the Phase II program.

## SECTION IV

### COST ANALYSIS

#### A. BACKGROUND

Depending on the process, regeneration includes some or all of the following:

- o Transferring the carbon to the regeneration reactor
- o The regeneration process itself
- o Transferring the carbon back to the adsorption vessel
- o Tail-gas purification
- o Purification/disposal of any solvents or chemicals used
- o Disposal of degraded carbon
- o Makeup for carbon losses.

In a detailed cost analysis, labor, chemical, and utility costs must be assigned to all these process steps. Also, the cost of maintenance, usually taken to be some fraction of the capital cost of the equipment, must be added to obtain the total operating costs.

Provided the size of the equipment is known, capital cost estimates can be obtained from vendors, published data for similar systems, or by using established costing procedures. The capital costs can then be amortized over a selected time period at the prevailing interest rate, and added to the operating and maintenance costs to obtain the total annualized cost of regeneration. The annual cost is normalized to the process stream that is being treated, for example, the amount of water that is stripped.

Other factors related to the regeneration process should be included in comparing the costs of different regeneration technologies. One such factor is the cost of enhancing the adsorption capacity of the carbon to reduce the frequency of regeneration. In the case of a stripping process, this would include the heating of the stripper off-gas to reduce its relative humidity.



Another factor is the cost of providing an additional adsorption vessel to take the place of a unit taken off-line for regeneration. The need for an extra vessel is determined by the size of the plant, the total number of adsorption vessels, the length of time a unit must be taken off line for regeneration, whether the process is continuous or semibatch, whether or not the regeneration is done in place, as well as the need for a standby unit. Because many of these factors are determined by plant characteristics, this cost has not been accounted for in the estimates presented here.

Because the intent here was merely to compare the cost of electric regeneration with more conventional steam regeneration, it is not necessary to estimate the cost of the stripping process, nor is it necessary to estimate the complete cost of regeneration. Only differential costs are required. Costs for components and operations that are equal or similar were not included in the analysis. This differential cost analysis was suggested by the Air Force during its evaluation of the Phase I proposal.

As mentioned in the last section, adequate data were not obtained in this investigation for microwave regeneration, so detailed costs estimates for this process are not included here. However, an indication of the approximate energy costs are given on the basis of published information.

Because both Rintoul and steam regeneration are to be carried out in place, transfer equipment and regeneration vessels are not required. Furthermore, the capital costs of steam-raising equipment are assumed to be at least as expensive as the cost of electrical insulation of the adsorption column and the electrodes required for electrical regeneration.

Based on the experimental data obtained in this Phase I program, the regeneration efficiency and carbon degradation appear to be at least as good as found for steam regeneration processes. The capacity of the carbon (and its activity) have been assumed to be not affected by the regeneration process, so the frequency of regeneration is the same for the two processes. Also, carbon losses are assumed to be the same. Consequently, a first approximation of the

differential cost can be made simply on the basis of the amount of steam and electric power required per unit mass of carbon.

Also, the cost of humidity control and tail-gas treatment, as appropriate to each regeneration process, is discussed qualitatively.

#### B. ELECTRICAL AND THERMAL ENERGY

Before proceeding with the cost estimates, a comparable cost for electricity and steam must be established. The tail gas issuing from the regeneration vessel was estimated to be:

- o At a temperature of about 180 °C or less (a relatively low grade heat),
- o Polluted with volatile organics.

The credit for the energy recovered from this stream was assumed to be either negligible, or off-set by the capital cost of heat exchangers and/or pollution control equipment required.

The basis for energy cost is a fuel price of \$0.006/MJ (\$6.40 per million Btu). For saturated steam at 10 atmospheres and 180 °C, having an enthalpy of  $2.8 \times 10^6$  J/kg, and assuming a boiler efficiency of 90 percent, the steam cost is:

$$\begin{aligned}\text{Steam cost} &= (2.8 \times 10^6 \text{ J/kg})(0.006 \times 10^{-6} \text{ \$/J})/0.9 \\ &= \$0.019/\text{kg} (\$8.6/1000 \text{ lb}) \\ &= \$6.40 \text{ per million BTU delivered.} \quad (14)\end{aligned}$$

Assuming a conversion efficiency of 33%, the electricity cost is:

$$\begin{aligned}\text{Electricity cost} &= (0.006 \text{ \$/MJ})(3.6 \text{ MJ/kWh})/0.33 = \$0.065/\text{kWh} \\ &= \$19.30 \text{ per million BTU delivered.}\end{aligned}\tag{15}$$

These rates are comparable to values used in the literature which generally include an allowance for recovery of capital. For example, Coutant (Reference 9) cites steam at \$10/1000 lb (\$7.40/million BTU) and electricity at \$0.08/kWh (\$23.70/million BTU), and Crittenden (Reference 5) uses \$5.00/1000 lb steam (\$3.70/million BTU) and \$0.055/kWh (\$16.30/million BTU).

#### C. ENERGY COSTS

Energy costs are estimated on the basis of 1 kg of carbon.

##### 1. Rintoul Regeneration

As discussed earlier, the average power consumed in the experiments was 40.7 watts for a period of 18 minutes. Using a value of 0.0572 kg for the mass of carbon in the column, the energy consumption for regeneration becomes:

$$ER = (0.0407 \text{ kW})(0.30 \text{ hrs}) / (0.0572 \text{ kg}) = 0.213 \text{ kWh/kg.}\tag{16}$$

This is equivalent to a heat input to the carbon of about 767 kJ/kg.

This energy consumption is compared to the theoretical requirement and with values cited in the literature. The specific heat of BPL carbon is 1.05 kJ/kg-K (Reference 5). Therefore, the theoretical energy required to raise its temperature through 160 °C (180 °C - 20 °C) is:

$$ET = (1.05 \text{ kJ/kg-K})(160 \text{ °K}) = 168.0 \text{ kJ/kg} = 0.0467 \text{ kWh/kg.}\tag{17}$$

Using a modified specific heat of 1.7 kJ/kg-K, which accounts for heat losses to the insulation and tube walls, etc. (Reference 21), the theoretical energy requirement increases to 272 kJ/kg (0.0756 kWh/kg). This is still less than half the value calculated for Rintoul heating.

Cole, et al. (Reference 19) report a value of 0.38 kWh/ kg dry carbon for Rintoul regeneration at 800 °C. Based on their graphical presentation of the carbon temperature in their 100 kg furnace as a function of time, the energy required to achieve a temperature of 180 °C was estimated to be approximately 20 percent of this value, or about 0.08 kWh/kg.

These comparable values indicate that the experimentally determined power consumption is of the correct magnitude and suggest that there might be scope for improved efficiency in a commercial-scale unit.

Using the calculated energy consumption, the cost of electricity for Rintoul regeneration is:

$$\text{ER\$} = (0.213 \text{ kWh/kg})(\$0.065/\text{kWh}) = \$0.014/\text{kg carbon.} \quad (18)$$

The volume of tail gas generated during the Rintoul process depends on the rate at which purge gas is supplied to the column. The optimum method of purging the desorbed organics was not determined in the Phase I investigation. During this investigation, little effect on the regeneration rate and power requirement was found on varying the air flow rate from 3 to 6 L/min. Using a value of 3 L/min and a time of 30 minutes, the purge gas rate for the Rintoul process is given by:

$$\text{QR} = (3 \text{ L/min})(30 \text{ min})/(0.0572 \text{ kg}) = 1573 \text{ L/kg.} \quad (19)$$

In the high-temperature Rintoul process (Reference 19), steam at a rate of less than 0.2 kg steam/kg carbon was used to purge the system. Using a steam density of 0.59 g/L, this rate converts to 340 L/kg, again indicating that the purge rates obtained in the test program are of the correct order, but can be improved upon.

## 2. Steam Regeneration

The energy consumed in steam regeneration is determined by the quantity of steam used. Steam rates reported in the literature vary from as high as 50 down to as little as 0.3 kg per kg carbon. Some values are summarized in Table 15.

TABLE 15. STEAM RATES FOR VOC REGENERATION.

Steam rate (kg/kg)	VOC	VOC Boiling Point (°C)	Remarks	Reference
50	PCE	121	At 170 °C, based on model calcs.	35
15-20	TCE	87	At 100 °C; determined by experiment	5
1-8	Various		Intermediate molecular weights	36
1.5	Various		At 100 - 140 °C; this value used in their economic analysis	9
0.3	MeCl <sub>2</sub>	-24		36

The steam rate required depends on the volatility of the compound being desorbed, and the steam temperature. With the exception of highly volatile methyl chloride, steam rates generally exceed 1 kg steam/kg carbon. We selected a low value of 1.5 kg/kg so as to provide a severe test of Rintoul economics. At this steam rate, the energy cost for steam regeneration is calculated as:

$$ES\$ = (1.5 \text{ kg/kg})(\$0.019/\text{kg}) = \$0.029/\text{kg carbon.} \quad (20)$$

Assuming no additional purge gas is used, the volume of tail gas is given by the steam rate:

$$QS = (1.5 \text{ kg/kg}) / (0.59 \times 10^{-3} \text{ kg/L}) = 2540 \text{ L/kg.} \quad (21)$$

#### D. COST COMPARISON

##### 1. Regeneration

###### a. Energy

On the basis of energy requirements for regeneration, operating costs for the Rintoul process are about half those for steam regeneration, even when using a very conservative steam rate of 1.5 kg steam/kg carbon. The steam rates of 15 - 20 kg/kg have been suggested for stripping TCE (Table 15). These high steam rates will result in operating costs of 20 times those for Rintoul heating. Also, the volume of tail gas produced in the Rintoul process is about 60 percent of that produced in steam regeneration, suggesting a further cost advantage. Furthermore, it is expected that when the system is scaled up the purge rates for the Rintoul process will be substantially reduced.

Even more dramatic is the comparison of Rintoul heating with off-site regeneration of GAC. The cost of off-site regeneration of GAC is \$1.50/lb carbon, or \$3.30/kg carbon. This is a little more than half of the price of GAC and about 225 times the operating cost of Rintoul regeneration.

###### b. Carbon Makeup

No significant carbon losses due to either loss of activity or other degradation were evident during the nine cycles completed on a single batch of carbon during the experimental investigation. Therefore, the carbon losses for Rintoul regeneration are tentatively assumed to be at least as low as for steam regeneration. Unfortunately, no information is available in the

literature on carbon losses during in-place Rintoul regeneration. More precise information will be obtained during the Phase II program.

c. Maintenance

Maintenance costs for the Rintoul system are anticipated to be mainly for electrode replacement. Maintenance of the electric distribution system is assumed to be negligible. On the other hand, maintenance costs for the case of steam regeneration can be expected to be higher because more process equipment is on site. This includes the boiler, the makeup water treatment system, the steam distribution system and, possibly, a fuel storage and supply system. Also, the very humid conditions during steam regeneration (saturated steam is normally used) can be expected to enhance potential corrosion problems in the adsorption vessel.

d. Labor

For reasons similar to those for maintenance, labor costs for the Rintoul process are also expected to be lower than for steam regeneration. Also, the electrical process should be easier to automate and control.

e. Capital

As discussed previously, capital costs for the two technologies can be assumed similar for the purpose of the approximate cost comparison made here. In fact, arguments could be made that the costs of a boiler, steam distribution system, and the tail-gas condenser required for steam generation will exceed those for electrical distribution, electrodes, and electric insulation of the adsorption vessel required for the Rintoul process. Furthermore, efficient and rapid Rintoul regeneration of GAC leads to a reduction in both the size of the adsorption vessel and the carbon inventory. This would make Rintoul regeneration even more attractive economically.

#### f. Economy of Scale

An important aspect of the Rintoul process relates to its application to small systems. No large cost overheads are involved, as for example in the case of a boiler for steam generation. All that is required is a source of electricity and a set of electrodes. Consequently, the system might be applied to small processes where economics would normally dictate off-site regeneration. Also, the equipment is portable, and might be applied to mobile systems.

Another important consideration relates to retrofitting existing systems. Costs should be minimal in that the only changes required are essentially the provision of an insulated lining and the electrodes.

#### 2. Humidity Control

Because the same method of heating the off-gas for humidity control can be used for either regeneration processes, this process step will not be more expensive for the Rintoul regeneration. However, preliminary results obtained during this program showed that low-level heating of the carbon during adsorption resulted in an increase in the GAC capacity as a result of humidity control. Further development of Rintoul heating technology might lead to additional cost savings for humidity control.

The heat transfer between the carbon and the gas stream was observed to be relatively poor. In general, the exit gas temperature did not rise above 36 °C (Table 14), even after the carbon had been at 150 °C or more for 20 minutes. This implies that the carbon can be heated, possibly in an intermittent fashion, to a temperature sufficient to desorb water, without significant loss of thermal energy to the gas stream. During the test program, the carbon bed was maintained at a temperature of 50 °C with a continuous power input of about 4 W. Provided the energy transferred to the gas stream is less than one-third of that used in directly heating the gas, the energy costs for Rintoul humidity control will be lower. Also, the capital cost of a heat exchanger will no longer be incurred.



### 3. Tail-Gas Treatment

For the same treatment process, this cost is governed essentially by the volume of tail gas generated. Tail-gas treatment for the Rintoul process, with its relatively low effluent flow rate, must be less than for steam regeneration.

Also, however, the characteristics of the tail gas are important in that they can affect the selection of the treatment technology. For example, the tail gas from Rintoul regeneration is relatively concentrated and dry (particularly if humidity control is applied during the adsorption cycle). Cooling this gas to recover the VOCs may therefore be feasible, and sale of the recovered solvents may be used to offset the treatment cost. In the case of steam regeneration, such a recovery step would be complicated by the lower concentration prevailing and because the condensate is essentially a polluted water stream. In one investigation, very little separate organic phase was formed on condensing the tail gas from steam regeneration (Reference 35).

Table 16 compares the costs of regeneration by Rintoul heating with steam and shows that Rintoul heating is an attractive option for in-place regeneration of GAC.

### 4. Microwave Regeneration

As stated, because of the nonuniformity in microwave heating, reliable data on which to estimate the cost of microwave regeneration were not generated. However, some data are available in the literature for VOCs adsorbed on dry carbon. Fang (Reference 27) regenerated 0.1 kg samples of carbons containing n-hexane, ethanol and acetone in a microwave at a power input of 252.6 W. Regeneration times (estimated from graphs in the paper) are 50, 18 and 12 minutes, respectively. The corresponding energy requirements are 2.1, 0.76 and 0.5 kWh/kg, which are higher than the 0.21 kWh/kg obtained for Rintoul regeneration.

TABLE 16. COMPARISON OF RINTOUL AND STEAM REGENERATION COSTS

<u>Operation</u>	<u>Relative Cost<sup>a</sup></u>	<u>Remarks</u>
Regeneration		
Energy	0.05-0.5	Assuming 1.5-15 kg steam/kg carbon.
Capital	<< 1	Electricity available on site; boiler and steam distribution system not required.
Humidity Control		
Energy	0.3	Assuming 4 °C rise for Rintoul heating and a 14 °C temperature rise with conventional means.
Capital	~0	No extra equipment required for Rintoul; heat exchanger required for conventional means.
Tail-Gas Treatment		
Energy	0.06-0.6	Based on purge gas flow rates.
Capital	< 1	Smaller reactor, much lower moisture content.

<sup>a</sup> Rintoul Regeneration Costs/Steam Regeneration Costs.

Fang (Reference 27) found that virtually no purge gas was required to desorb the organics during microwave regeneration. Tail-gas cleaning may therefore be less expensive than with Rintoul heating.

Capital costs will be higher to the extent that the microwave "oven" is more expensive than the electrodes and electric insulation for the Rintoul process.

#### 5. Other Processes

Much of the literature data for energy consumption for both thermal and electrical regeneration processes are for aqueous systems. In these processes, regeneration occurs at elevated temperatures in a separate furnace, and a significant portion of the energy is consumed for drying the carbon. These data are not suitable for comparison with the low-temperature Rintoul process under consideration.

One process that might be of interest is Airco's hot nitrogen process, which is claimed to have operating costs up to 50 percent lower than for steam (Reference 10), and therefore might have costs comparable to Rintoul regeneration. The heat exchanger used to heat the stripper off-gas for humidity control may serve also to heat the nitrogen in the regeneration step. Nevertheless, a source of heat, either steam or flue gas, must again be available on site. If electrical heating is used, it might as well be applied directly to the carbon as in the Rintoul process.

## SECTION V

### CONCLUSIONS

Rintoul regeneration is a commercially proven process that has been applied to high temperature regeneration of liquid-phase GAC in special purpose furnaces. The investigation conducted here has demonstrated that the Rintoul process can be efficiently applied to low-temperature in-place regeneration of vapor-phase GAC.

Repeated loading and regeneration of the carbon column showed no significant loss of adsorption capacity. The GAC maintained its capacity after successive regenerations with Rintoul heating. This indicates that the organics are efficiently desorbed and that there is little loss in activity due to oxidation of the carbon as might have occurred, for example, had non-uniform heating resulted in excessive temperatures.

Adsorption tests were carried out at high-influent TCE concentrations to obtain data for many adsorption-regeneration cycles. The vapor-phase GAC capacities obtained at these high influent concentrations with regeneration by Rintoul heating were consistent with other published data at lower TCE concentrations with more conventional methods of regeneration. Similar results would be expected to be obtained at lower TCE concentrations.

Low-level heating of the GAC during adsorption counteracted the deleterious effect of humidity on TCE capacity and provided a method of decreasing the capacity of GAC for water faster than the TCE capacity. This method is an alternative to heating the influent gas to reduce the humidity.

Capacities measured during regeneration were not consistent with the capacities measured during adsorption. The measured regeneration capacities were inaccurate primarily because condensation was occurring in the sampling lines, reducing the total TCE detected by the gas chromatograph.

Power was supplied to the carbon column during regeneration. The maximum temperature in the carbon bed ranged from a high of 188 °C to a low of 127 °C. High power resulted in higher carbon bed temperatures. The energy adsorbed by the air stream during regeneration was very small compared to the energy adsorbed by the activated carbon.

Regeneration was also carried out using microwave heating. The microwave oven gave nonuniform heating over the length of the column so that not all of the TCE came off during desorption. Because of programmatical constraints, further work on microwave heating was not carried out.

Two methods of collecting TCE in a concentrated form were successfully tested. Both processes involved recovery of the TCE in a condensate. Further work is being planned for a Phase II program.

Energy costs for the Rintoul process are conservatively estimated to be about half that for steam regeneration. All other costs, including labor, tail-gas purification, maintenance, and capital, are also expected to be less than those for steam. A comparison of the cost of the Rintoul process with other methods of dealing with VOC's in airstreams, especially the use of catalytic oxidation, is planned to be made as part of a Phase II program.

Apart from its favorable costs, other major advantages of Rintoul regeneration are the ease of retrofitting, its application to small systems, and its portability.

Based on literature data, energy costs for microwave regeneration are slightly higher than for the Rintoul process. The amount of purge gas required may be less than required in Rintoul regeneration.

## SECTION VI

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